

**ASSESSMENT OF HEAVY METAL CONTAMINATION  
AND ITS MOBILIZATION FROM SELECTED LANDFILLS  
IN SELANGOR**

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**FACULTY OF SCIENCE  
UNIVERSITY OF MALAYA  
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**2017**

**ASSESSMENT OF HEAVY METAL  
CONTAMINATION AND ITS MOBILIZATION FROM  
SELECTED LANDFILLS IN SELANGOR**

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**DISSERTATION SUBMITTED IN PARTIAL  
FULFILMENT OF THE REQUIREMENTS FOR THE  
DEGREE OF MASTER OF TECHNOLOGY  
ENVIRONMENTAL MANAGEMENT**

**INSTITUTE OF BIOLOGICAL SCIENCES  
UNIVERSITY OF MALAYA  
KUALA LUMPUR**

**2017**

**UNIVERSITY OF MALAYA**  
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## ABSTRACT

In this study, the comparison of the total concentration and mobilization of heavy metals (Pb, Zn, Mn, Cu and Ni) in soil from active and closed cells in Jeram Sanitary Landfill as well as the contamination factor and risk of heavy metals in soils were investigated. Soil samples from active and closed cell were dried and homogenized and sequentially extracted. The total heavy metal concentration were determined by pseudo total digestion procedure, while the speciation pattern of heavy metals were done using Tessier's five steps sequential extraction procedure and further analyzed with ICP-MS. The results obtained from the analysis indicated that the concentration of heavy metals in open cell were higher than that from the closed cell. Level of Pb concentration in the active cell was higher than the closed cell in all samples. In a sequential extraction procedure, it is obvious that Mn was identified in the greatest amounts in mobile phase whereas Cu and Pb showed the greatest amount in immobile phase in soils at all stations. The heavy metal contamination factor was also determined in this study, based on the contamination factor values to indicate the degree of heavy metals risk to the environment. It was found that the soil was possibly polluted with Mn, Zn and Ni. RAC value calculated in this study showed medium risk for active cell and low risk for closed cell for most of the heavy metals except for Mn. Although Pb was found to be the highest concentration in all samples due to its non-mobility state in soil, it is at low risk to the environment.

## ABSTRAK

Dalam kajian ini, perbandingan jumlah kepekatan dan pergerakan logam berat (Pb, Zn, Mn, Cu dan Ni) di dalam tanah dari sel aktif dan tertutup di Tapak Pelupusan Sanitary Jeram serta faktor pencemaran dan risiko logam berat dalam tanah telah dikaji. Sampel tanah dari sel aktif dan tertutup telah di keringkan dan diekstrakan secara berturutan. Jumlah kepekatan logam berat ditentukan dengan prosedur jumlah prosedur pencernaan, manakala corak penspesiesan logam berat telah dilakukan dengan menggunakan prosedur pengekstrakan lima langkah Tessier yang berurutan dan seterusnya dianalisis dengan ICP-MS. Keputusan yang diperolehi daripada analisis menunjukkan bahawa kepekatan logam berat dalam sel terbuka adalah lebih tinggi daripada yang dari sel tertutup. Tahap kepekatan Pb dalam sel aktif adalah lebih tinggi daripada sel tertutup dalam semua sampel. Dalam prosedur pengekstrakan berurutan, ia adalah jelas bahawa Mn telah dikenal pasti dalam jumlah yang besar dalam bentuk fasa bergerak manakala Cu dan Pb menunjukkan jumlah yang paling besar dalam fasa tidak bergerak dalam tanah di semua stesen. Faktor pencemaran logam berat juga telah ditentukan dalam kajian ini, berdasarkan nilai faktor pencemaran untuk menunjukkan tahap risiko logam berat risiko kepada alam sekitar. Telah mendapati bahawa tanah adalah mungkin tercemar disebabkan Mn, Zn dan Ni. Nilai RAC dikira dalam kajian ini menunjukkan risiko sederhana untuk sel aktif dan berisiko rendah untuk sel tertutup bagi kebanyakan logam berat kecuali Mn. Walaupun Pb didapati mempunyai kepekatan tertinggi dalam semua sampel, walaubagaimanapun di sebabkan sifatnya yang tidak bergerak dalam tanah, ianya berisiko rendah kepada alam sekitar.

## ACKNOWLEDGEMENTS

In the name of Allah, the Most Gracious and the Most Merciful. Alhamdulillah, all praises to Allah for the strength and His blessing in completing this thesis. This opportunity constantly improves me in developing my career especially in this field of environment management and chemical analysis. I cannot express enough thanks to my supervisor for her continued support and encouragement, Dr Sharifah Mohamad. I offer my sincere appreciation for the learning opportunities provided by my supervisor.

Furthermore, I would like to take this opportunity to express my gratitude to my co supervisor Dr. Fauziah and her research group for the support, valuable information and guidance which helped me in completing this task through various stages. My completion of this project could not have been accomplished without the support of my family and friends.

Lastly, to Ms. Natasha Shafeez, Ms. Eleena Norsin for their constant encouragement and without them this thesis would not be possible. Thank you for understanding and sacrificing time helping me with patience and always gave me moral support and motivation.

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## LIST OF SYMBOLS AND ABBREVIATIONS

ACROS	:	Automated Cargo Release & Operations Service
APHA	:	American Public Health Association
ASTME	:	American Society of Tool and Manufacturing Engineers
DOE	:	Department of Environment
E	:	East
g	:	gram
H <sub>2</sub> O <sub>2</sub>	:	Hydrogen peroxide
HCl	:	Hydrochloric acid
HNO <sub>3</sub>	:	Nitric Acid
HOAc	:	Acetic acid
hr	:	Hour
ICP-MS	:	Inductively coupled plasma-mass spectrometry
ISO	:	International Organization for Standardization
M	:	Molar
mg/L	:	milligram per liter
MgCl <sub>2</sub>	:	Magnesium Chloride
min	:	Minute
mL	:	millilitres
N	:	North
NaOAc	:	Sodium acetate
NH <sub>3</sub> OHCl	:	Hydroxyl ammonium chloride
NH <sub>4</sub> OAc	:	Ammonium acetate
RAC	:	Risk assessment code
rpm	:	Revolution per minute

sec	:	Second
U.S.EPA	:	United State of Environmental Protection
V	:	Voltage
W	:	Walt
WHO	:	World Health Organization
%	:	Percentages
<	:	Less than
>	:	More than
°C	:	Degree Celsius
$\Omega/cm$	:	Ohm per centimeter

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## CHAPTER 1: INTRODUCTION

### 1.1 Heavy Metals Pollution from Landfill

The increase in the number of human and industrial activities has led to massive industrialization and urbanization (Ahmad *et al.*, 2009). Various industrial and urban activities have resulted in the increment of waste being generated. Besides that, it also brings with it some disadvantages for instance an increase in the introduction of toxic heavy metals into the environment which would result in negative environmental impacts. Heavy metals contamination to the environment is a serious and global problem (Jiang *et al.*, 2013).

The presence of toxic heavy metals especially in the landfills may create an acute pollution of soil and water and also may pose health hazards to the people. Solid waste containing toxic heavy metal particularly in landfills are generated from various sources such as agricultural, industrial as well as residential and commercial activities such as electronic wastes, painting wastes, used batteries and others as illustrated in Figure 1.1 (Agamuthu & Nagendran *et al.*, 2010). Municipal Solid Waste (MSW) generation in Malaysia has exceeded 19,000 daily and 30,000 tonnes by the year 2011 (Agamuthu & Fauziah, 2011). However, in recent study by Intan (2015), Malaysia has recorded an increase in solid waste generation in 2012 by 58% (33,000 tonnes per day) as compared to the year 2009. The ever increasing waste generation has resulted in the release of waste containing toxic heavy metal into adjacent environment and eventually causing a serious threat towards environment and human health (Donald *et al.*, 2010).



**Figure 1.1:** Various of waste in landfill (Source: National Geographic 2008)

Time after time, soil in landfill received potentially toxic elements from both natural and anthropogenic activities including waste dumping (Ali *et al.*, 2014). Contamination of heavy metals in soil may potentially occur at old landfill sites that received industrial wastes and other substances that may produce heavy metals (USDA NRCS, 2000). However, according to Karim *et al.* (2014) the effects of heavy metals are found to differ with the situations prevailing in the landfill and its binding forms. The active cell (landfill) that exposed to the atmospheric condition undergoes different effects due to oxygen diffusion where heavy metals are easily available and released. As a result, the increasing amount of waste in landfill has created a major ecological concern for the environment and human health (Ekanem *et al.*, 2013).

According to the United States Department of Agriculture, Natural Resources Conservation Services (USDA NRCS) (2000), metals in soil were retained from a lot of activities, mainly toxic anthropogenic activities (i.e. manufacturing, mining, agriculture, utilizing of synthetic products like paints, industrial wastes and pesticides) and a few

from natural activities which is rarely occurring at toxic levels. As a matter of fact, soil contamination from anthropogenic sources can affect the natural ecosystem in the world since the contamination occurs when the soil composition differs from the normal composition (Shayler *et al.*, 2009). Indeed, soil pollution can be understood by the presence of some constituent in the soil resulted from the human activity, at certain concentration that there is a potential significant risk towards to users of the soil (Sparks *et al.*, 2003). The risk can be categorized in many forms for example, impairment health for human, animals or plants. Also, contaminants may not be classified as pollutants unless they have some harmful effect to living organisms (Manta *et al.*, 2002).

Waste composition varies from one source to another depending on the type of industries ranging from manufacturing industries to household activities. The waste that contain high concentrations of heavy metals include food waste (Cu, Cr, Pb, Zn), plastics (Cd, Cu, Pb, Ni, Zn), coal cinders (Cu, Cr, Zn), glass (Cd, Cr, Ni, Zn), dust (Cu, Cr, Ni), and textile (Cu, Pb, Ni). The soil problem is worsened by the fact that many landfills lack of bottom liner and or collection system of leachate. This increase the possibility of dissipation of leachate through the landfill layers to contaminate the soil. On the other hand, the migration of leachate resulted from waste dumping could also lead to the contamination of soil in landfill (Yadav *et al.*, 2010).

Excessive accumulation of heavy metals in soils could lead to toxicity amongst humans and animals through plant uptake and human consumption (USDA NRCS, 2000). Due to excessive levels of heavy metals accumulated in soil, it may also jeopardize groundwater quality in which the chemicals and heavy metals may transfer from one environment to another. Hence, the prevalence of heavy metals in soil (depending on the concentration) is an environmental and public health concern. Table 1.1 summarizes some of the possible chronic effects of several heavy metals towards human health.

**Table1.1:** Possible Chronic Health Effects from Selected Heavy Metals (USDA NRSC, 2000)

<b>Heavy Metals</b>	<b>Possible Chronic Effect</b>
Lead	Mental lapse
Cadmium	Affects kidney, liver and GI tract
Arsenic	Skin poisoning, affects kidneys & central nervous system
Manganese	Nervous system, lower visual reaction time, poorer hand steadiness, and impaired eye-hand coordination
Nickel	Respiratory effects, including a type of asthma specific to nickel, decreased lung function, and bronchitis
Copper	Damage the liver and kidneys

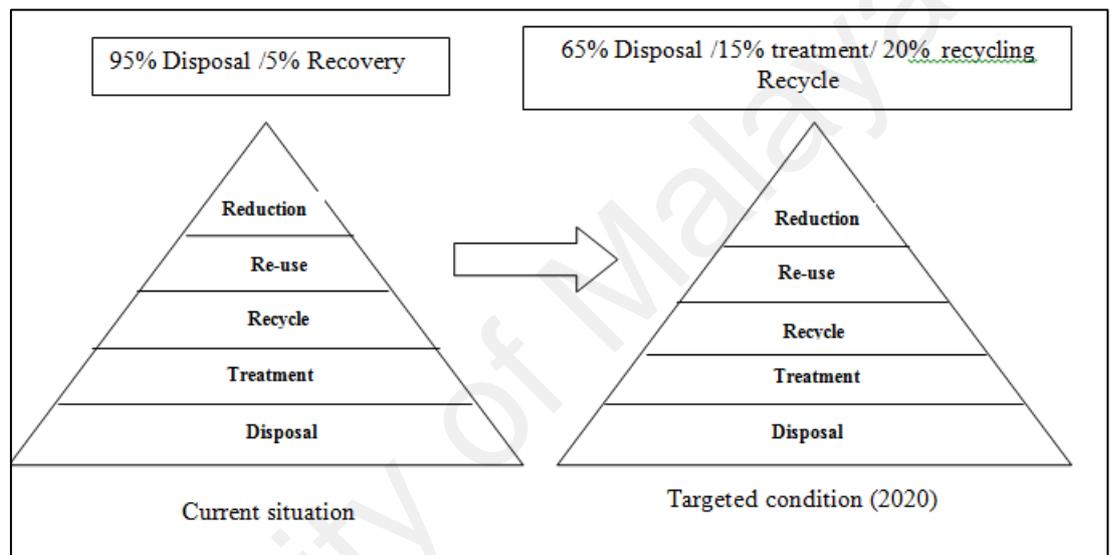
In spite of the increasing active urban development and industrialization in Malaysia, waste management is relatively poor (Sreenivasan, 2012). Therefore, it has been a major public health and environmental importance in many countries including Malaysia as it may introduce danger to health and safety of the public. According to McAllister (2015), Hashim (2012) and Agamuthu & Fauziah (2010), failure in waste management would lead to detrimental effects on the environment.

In order to ensure continuous preservation of the environment, a proper waste management is essential to reduce potential pollution from landfill. A proper waste management encompasses all the activities associated with the control of generation, storage, collection, transportation, processing or treatment, as well as, disposal of waste consistent with the best practices of public health, economics and finance, engineering, administration, legal and environmental considerations (Johari *et al.*, 2014 and Sreenivasan *et al.*, 2012).

The growing concern over the need for a proper management of solid waste in Malaysia has prompted the Government to establish a comprehensive waste management and disposal system. The government has also taken comprehensive steps to mitigate waste problem by developing and implementing appropriate laws and guidelines (Department of Environment, 2010). Currently, the management and disposal of waste in Malaysia is controlled and guided by the Solid Waste and Public

Cleansing Management Act (SWPCMA), 2007 together with the Environmental Quality (Control of Pollution from Solid Waste Transfer Station and Landfill) Regulations, 2009 (Ministry of Housing and Local Government, 2005).

In Malaysia, landfill disposal is the most common option of waste management. The following Figure 1.2 illustrates the comparison between current and the targeted condition of waste management hierarchy in Malaysia (MHLG, 2005).



**Figure 1.2:** Malaysian current and targeted waste hierarchy by 2020 (MHLG, 2005)

## 1.2 Problem Statement

Landfills pose a big problem to the environment in which different kinds of hazards are produced especially heavy metals. It can cause serious pollution when it gets in contact with the surrounding soil, surface water, and groundwater leading to detrimental effects to living organisms (Al Raisi *et al.*, 2014). Many studies proved that several impacts from improper landfilling activities include, leachate contamination to surface and groundwater (Fauziah *et al.*, 2005), release of landfill gases such as methane (40–50%) and carbon dioxide (50%) (Agamuthu, 2001), infestation of pest (Ojeda-Benitez

*et al.*, 2003) and in most cases accumulation of heavy metals in soil (Balkhair *et al.*, 2016).

The amount of waste generated in Malaysia is increasing with daily amount of solid wastes produced has reached between 0.5-2.5 kg per capita per day (and a total of 25000-30000 tons per day) (Johari *et al.*, 2014 and Fauziah & Agamuthu, 2012). Thus, the escalated amount of waste in landfill has created a major ecological concern for the environment and human health (Ismail & Manaf, 2013 and Budhiarta *et al.*, 2012).

Various sources of waste disposed in landfills for example, food waste, industrial and domestic waste and agricultural waste had contributed to the leaching of different types of heavy metals in soil (Wuana & Okieimen, 2011). The build-up of heavy metal in soils (depending on the concentration) is considered an environmental concern. Heavy metals in soil would directly impacted human health because the uptake of heavy metals by plants and subsequent accumulation along the food chain is a potential threat to animal and human health (Liu *et al.*, 2013; Nannoni *et al.*, 2011; Sprynskyy *et al.*, 2011; Singh and Kalamdhad, 2011 and O'Connell *et al.*, 2008). Furthermore, leachate from landfills containing heavy metals could lead to the contamination of groundwater and surface water when it dissipate through soil and eventually causing detrimental effect to living organisms (Al Raisi *et al.*, 2014).

Numerous studies were conducted on heavy metals contamination in many soil types, but fewer had focused on landfill soil (active and closed). In addition, due to lack of information on speciation of heavy metal in landfills and its risk to the environment, this current study intended to determine the risk associated with heavy metal contamination in soil and to understand the behaviour of selected heavy metal in term of its mobilization in soil.

### 1.3 Scope of Study

In this study, the determination of total concentration and its mobilization of heavy metal such as Mn, Ni, Zn, Cu and Pb in soil from active and closed cell of sanitary landfill were done. These five elements were selected since these are the most commonly found in domestic waste (Al Raisi et al., 2014; Chibuike & Obiora, 2014; Kanmani & Gandhimathi, 2013 and Bahaa-Eldin et al., 2008). In addition, these five elements were reported as the heavy metal of environmental and health concern. The soil samples were taken from two different types of cells; namely, active and closed cells from a sanitary landfill in order to compare the differences of total heavy metal concentration and heavy metals mobilization in soil. For example, soils from active cell are more exposed to the environmental changes such as sunlight and rainfall which may influence the soil condition chemically. On the other hand, soil from the closed cell has lining material which prevents much intrusion to the reaction that occurs below. In addition, soil from closed cells have become inactive therefore, no additional chemical reaction from surroundings. The risk of heavy metals contamination was also conducted as soil from closed cell is estimated to have less risk when compared to the soil from active cell. This is because active cells still received various types of wastes which contain different types of heavy metals thus producing more risks. Whereas, the risk of heavy metals contamination in closed cell only comes within its surrounding without interference from external factors. Overall, the differences in the characteristic of both active and close cells will show varying outcomes in terms of heavy metal contamination and mobilization in soil. In addition, it is very important to understand how the nature and movement of heavy metals differ in both cells. Besides that, this comparative study is relatively new in Malaysia. Therefore, the results of this study are expected to contribute to the existing database on heavy metal contamination in landfill

and other related studies. The results also likely serve as a baseline data for future research.

#### **1.4 Research Objectives**

The study was carried out with the following objectives:

1. To compare the total concentration of heavy metals in soil from active and closed cells in sanitary landfill.
2. To compare the mobilization of heavy metals in soil from active and closed cells in sanitary landfill.
3. To determine the contamination factor and to conduct risk assessment for heavy metals in soils collected from active and closed cells in sanitary landfill.

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## CHAPTER 2: LITERATURE REVIEW

### 2.1 Definition of Heavy Metals

Numerous amount of toxic pollutants discarded into the environment have represented huge sinks of heavy metals ever since the introduction of Industrial Revolution (Forstner & Wittmann, 2012). Heavy metals are natural constituents of the earth's crust, but the indiscrimination of human activities has drastically altered their geochemical cycles and biochemical balance (Singh *et al.*, 2011).

Heavy metals are the metallic elements that have high or at least five times greater than water in terms of its atomic weight and a density with above 7 g/cm<sup>3</sup> (Duruibe *et al.*, 2007). For example mercury, chromium, cadmium, arsenic, and lead which can destruct living things at low concentrations and able to accumulate in the food chain (United States Environmental Protection Agency, 2015; Tchounwou *et al.*, 2012). According to Duffus (2002) the term “heavy metals” has been broadly used as a group name for metals and semimetals (metalloids) that have been related with contamination and potential toxicity.

In the world of environmental remediation, heavy metals are typically refers to one or more elements that may exist at toxic waste dumping sites. Heavy metals are often caused the greatest risk due to their toxicity or the present of high concentration. Apart from that, it is also known as trace elements since their presence in trace concentration in numerous environmental media. Moreover, the toxicity of heavy metal depends on many factors such as dose, chemical species, route of exposure, and duration and frequent of exposure (Heudorf *et al.*, 2007). On the other hand, the physical factor such as temperature, adsorption and phase association, as well as, the chemical factors, such as lipid solubility, complexation kinetics and water partition give major influence on the bioavailability of heavy metals (Chibuike *et al.*, 2014). According to World Health

Organization (WHO), 2011 cadmium (Cd), mercury (Hg), lead, (Pb), arsenic (As), iron (Fe), copper (Cu), zinc (Zn), cobalt (Co), manganese (Mn), and nickel (Ni) are listed as ten metals of major public concern.

Heavy metals negatively affect the environment and human's health (Robinson *et al.*, 2009). This is because heavy metals cannot be degraded nor destroyed and they are persistent in all environmental media. Heavy metals are naturally occurring in the environment; apart from that rapid growth of anthropogenic activities has also contributed to the elevation of heavy metals in the environment (Akan *et al.*, 2013). The activities include burning of fossil fuels, enhancement of heavy industries, etc. that may lead to pollution of air, water especially the surface water and groundwater and soil (Mapanda *et al.*, 2005). Figure 2.1 shows disposal of toxic waste resulting from anthropogenic activities (i.e. heavy industries).



**Figure 2.1:** Disposal of toxic waste (Source: National Geographic, 2016)

## 2.2 Sources and Effects of Heavy Metals in Environment

Heavy metal pollutions can arise from many sources. Nowadays, most of heavy metals were introduced into the environment through natural or anthropogenic sources (Duruibe *et al.*, 2007). As reported by Khillare *et al.* (2015), sources of heavy metals in the environment include the geogenic, domestic effluents, industrial, agricultural, pharmaceutical and atmospheric sources. Two common sources of heavy metals pollution to the environment are point sources and non-point sources.

According to the Department of Environment (DOE), Malaysia (2016), a point source is defined as pollution that can be readily identified from a specific source such as pollution from factory or treatment works. On the other hand, a pollution origin which cannot be specifically defined and mainly diffused for instance agricultural activities or surface runoff is known as non-point source (Pekey *et al.*, 2015).

In terms of point source pollution of soil contamination, soils may become contaminated by the accumulation of heavy metals through emissions from the rapidly expanding industrial areas, mine tailings, disposal of high metal wastes, leaded gasoline and paints, land application of fertilizers, animal manures, sewage sludge, pesticides, wastewater irrigation, coal combustion residues, spillage of petrochemicals, and atmospheric deposition (Zhang *et al.*, 2011 and Khan *et al.*, 2008). Soils in landfill are known as one of the major sink for heavy metals released into the environment. The major sources of heavy metals in landfills are the co-disposed industrial wastes, incinerator ashes, mine wastes and household hazardous substances such as batteries, paints, dyes, inks, etc. (Erses & Onay, 2003).

Solid waste disposals (open dumps, landfills, sanitary landfills or incinerators) represent a significant source of metals released into the environment (Rizo *et al.*, 2012; Bretzel & Calderisi, 2011; Iwegbue *et al.*, 2010; Waheed *et al.*, 2010 and Yarlagadda *et al.*, 1995). Poor waste management poses a great challenge to the well-being of city

residents, particularly those living adjacent the dumpsites due to the potential of the waste to pollute water, food sources, land, air and vegetation. On the other hand, improper disposal and handling of waste can also leads to environmental degradation, destruction of the ecosystem and poses great risks to public health (Ahmed *et al.*, 2011). In addition, lands near landfills are prone to heavy metal contamination by various wastes containing hazardous compound. Throughout the time, leachate produced from discarded wastes will infiltrate into the ground, seeping into surface and groundwater system and eventually result in water pollution (Gwenzi et al., 2016). An excessive discharge of heavy metal into the environment is a critical environmental concern and poses an adverse impact to public health and safety (Agamuthu & Fauziah, 2010).

The distribution of heavy metals in the environment varies from place to place. The following section highlights the sources and effect of several heavy metals to environment and human health:

**a) *Lead (Pb)***

According to USEPA (2015), lead is a naturally occurring element found in small amounts in the earth's crust. While it has some beneficial uses, it can be toxic to humans and animals. Lead in environment can also negatively affect the human health. Lead can be found in all parts of our environment – the air, the soil, the water, and even inside our homes (Heudorf et al., 2007). Much of our exposure comes from human activities including the use of fossil fuels including past use of leaded gasoline, some types of industrial facilities, and past use of lead-based paint in homes (Gordon et al., 2002). Lead and its compounds have been used in a wide variety of products found in and around our homes, including paint, ceramics, pipes and plumbing materials, solders, gasoline, batteries, ammunition, and cosmetics. These usually ended up disposed being in landfill.

It is known that lead accumulates in the soil, particularly soil with a high organic content (Greene, 2014; Finster *et al.*, 2004). Lead deposited on the ground is transferred to the upper layers of the soil surface, where it may be retained for up to 2000 years (Greene, 2014 and Siccama *et al.*, 1978). In undisturbed ecosystems, organic matter in the upper layer of soil surface retains atmospheric lead. Atmospheric lead in the soil will continue to move into the micro-organism and grazing food chains, until equilibrium is reached.

Given the chemistry of lead in soil, Johansson (2001) suggests that the uneven distribution of lead in the ecosystems can displace other metals from the binding sites of organic matters. Lead may hinder the chemical breakdown of inorganic soil fragments and lead in the soil may become more soluble, thus being more readily available to be taken up by plants. Plants on land tend to absorb lead from the soil and retain most of the element in their roots. There is some evidence (Sharma *et al.*, 2005) that plant foliage may also take up lead and it is possible that this lead is moved to other parts of the plant. Some species of plant have the capacity to accumulate high concentrations of lead (Howe *et al.*, 2002).

High levels exposure from lead to human can damage almost all organs and organ systems, most importantly the central nervous system, kidneys and blood, and death at excessive levels (Tong *et al.*, 2000). At low levels, haem synthesis and other biochemical processes are affected while, psychological and neurobehavioral functions are impaired (Al-Terehi *et al.*, 2015). There is a range of other effects for instance; lead can cause damage to the kidneys, liver, brain and nerves, and other organs. Exposure to lead may also lead to osteoporosis (brittle bone disease) and reproductive disorders (Flora *et al.*, 2012).

Besides that, lead could affect the brain and nerves (Hsiang *et al.*, 2011). Excessive exposure to lead causes seizures, mental retardation, behavioural disorders, memory problems, and mood changes (Dubovický, 2010). Low levels of lead damage the brain and nerves in foetuses and young children, resulting in learning deficits and lowered IQ (WHO, 2016). In addition, lead exposure causes high blood pressure and increases heart disease, especially in men (Navas-Acien *et al.*, 2007).

Lead exposure may also lead to anaemia, or weak blood (Gordon *et al.*, 2002). On the other hand, lead could also potentially give adverse effect to the environment. Wild and domestic animals can ingest lead while grazing. They experience the same kind of effects as people who are exposed to lead. Low concentrations of lead can slow down vegetation growth near industrial facilities. Lead can also enter water systems through runoff and from sewage and industrial waste streams. Elevated levels of lead in the water can cause reproductive damage in some aquatic life and cause blood and neurological changes in fish and other animals (Solomon *et al.*, 2008).

**b) Manganese (Mn)**

Manganese is naturally ubiquitous in environment (Vieira *et al.*, 2012). Manganese is a very common compound that can be found everywhere on earth. Manganese compounds exist naturally in the environment as solids in the soils and small particles in the water. Manganese particles in air are present in dust particles. These usually settle to earth within a few days. Humans enhance manganese concentrations in the air by industrial activities and through the burning of fossil fuels. Manganese from human sources can also enter surface water, groundwater and sewage. Through the application of manganese pesticides, manganese will enter soils (Gavrilescu, 2005). Manganese is use primary in steel production to improves hardness, stiffness and strength. According to Al-Raisi *et al.* (2014), Mn in landfill proves to originate from the disposal of

considerable amount of steel. In addition, Mn can be related to the unregulated disposal of old batteries (Abu-Daabes *et al.*, 2013).

Manganese is one out of the three toxic essential trace elements. It is toxic when present at too high concentrations in a human body (Singare *et al.*, 2012a). The uptake of manganese by humans mainly takes place through food, such as spinach, tea and herbs. The foodstuffs that contain the highest concentrations are grains and rice, soya beans, eggs, nuts, olive oil, green beans and oysters (Zaidan *et al.*, 2013). After absorption in the human body manganese will be transported through the blood to the liver, the kidneys, the pancreas and the endocrine glands (Mohan *et al.*, 2008). Manganese effects occur mainly in the respiratory tract and in the brains (Levy *et al.*, 2013). Symptoms of manganese poisoning are hallucinations, forgetfulness and nerve damage. Manganese can also cause Parkinson, lung embolism and bronchitis (Mohan and Sreelakshmi, 2008). When men are exposed to manganese for a longer period of time they may become impotent (Kukiattrakoon *et al.*, 2010).

For animals, manganese is an essential component for over 36 enzymes that are used for the carbohydrate, protein and fat metabolism. In animals with too little manganese, interference to normal growth, bone formation and reproduction will occur (Soldin and Aschner, 2007). For some animals the lethal dose is quite low, which means they have little chance to survive even smaller doses of manganese exceed the essential dose (Soldin and Aschner, 2007). Manganese can cause lung, liver and vascular disturbances, declines in blood pressure, failure in development of animal foetuses and brain damage (Jaishankar *et al.*, 2014). When manganese uptake takes place through the skin it can cause tremors and coordination failures (Quremi and Ayodele, 2014). Finally, laboratory tests with test animals have shown that severe manganese poisoning would be able to cause tumour development in animals (Nadzirah *et al.*, 2010).

In plants, manganese ions are transported to the leaves after the uptake from soils. When too little manganese can be absorbed from the soil, this causes disturbance in plant mechanisms (Millaleo *et al.*, 2010). Manganese can cause both toxicity and deficiency symptoms in plants. When the pH of the soil is low manganese deficiencies are more common (Foy, 1984). Highly toxic concentrations of manganese in soils can cause swelling of cell walls, withering of leaf and brown spots on leaves (Singare *et al.*, 2012b). Deficiencies can also cause these effects. Between toxic concentrations and concentrations that cause deficiencies a small area of concentrations for optimal plant growth can be detected (Vose *et al.*, 1982).

**c) Zinc (Zn)**

Zinc is an element commonly found in the Earth's crust. Naturally, there are variety of foods contain zinc for example, oysters contain more zinc but red meat and poultry provide the majority of zinc to human (Maret and Sandstead, 2006). Other good sources include beans, nuts, and certain types of seafood, whole grains, fortified breakfast cereals, and dairy products (US Department of Agriculture, 2000).

Human activities may contribute to the abundant of Zn in the environment. Releases of Zn from anthropogenic sources exceed the release from natural sources (Tchounwou *et al.*, 2012). The greatest sources of zinc in the environment are probably from the soil. These sources are related to mining and metallurgic operations involving zinc; and use of commercial products containing zinc which resulted from the disposal of batteries, fluorescent lamps, food waste, and burning tires in the landfill (Fekiacova *et al.*, 2015).

Zinc is a trace element that is essential for human health. When people has too little zinc in their body they can experience a loss of appetite, decreased sense of taste and smell, slow wound healing and skin sores (Walravens, 1979). Zinc-shortages can even cause birth defects (Raju and Naidu, 2013). Although humans can handle proportionally

large concentrations of zinc, too much zinc can still cause eminent health problems, such as stomach cramps, skin irritations, vomiting, nausea and anemia (Bojic et al., 2009). Higher levels of zinc can damage the pancreas and disturb the protein metabolism, and cause arteriosclerosis (Oyaro *et al.*, 2007). Extensive exposure to zinc chloride can cause respiratory disorders (Ihedioha *et al.*, 2014).

In the work place, environment zinc contagion can lead to a flu-like condition known as metal fever (Al-Teheri *et al.*, 2015). This condition will pass after two days and is caused by over sensitivity. Zinc can be a danger to unborn and new-born children. When their mothers have absorbed large concentrations of zinc the children may be exposed to it through blood or milk of their mothers (Wassermen *et al.*, 2006).

The world's zinc production is still rising. This basically means that more and more zinc ends up in the environment. Water is polluted with zinc, due to the presence of large quantities of zinc in the improper treated industrial wastewater (Bojic et al., 2009). One of the consequences is that rivers are depositing zinc-polluted sludge on their banks. Zinc may also increase the acidity of waters (Raut *et al.*, 2012). Water-soluble zinc that is located in soils can contaminate groundwater (Wu *et al.*, 2010).

Some fish can accumulate zinc in their bodies, when they live in zinc-contaminated waterways. When zinc enters the bodies of these fish it is able to biomagnify up the food chain. On zinc-rich soils only a limited number of plants have a chance of survival. That is why there is not much plant diversity near zinc-disposing factories. Zinc can interrupt the activity in soils, as it negatively influences the activity of microorganisms and earthworms. The breakdown of organic matter may seriously slow down because of the presence of zinc (Chukwuma *et al.*, 2010). Plants often have a zinc uptake that their systems cannot handle, due to the accumulation of zinc in soils. The effects upon plants zinc is a serious threat to the productions of farmlands. Yet, of this zinc-containing manures are still applied (Chirila *et al.*, 2008).

**d) Nickel (Ni)**

Nickel is a naturally occurring constituent that exist in numerous mineral forms. Natural sources of atmospheric nickel comprise of wind-blown dust, derived from the weathering of rocks and soils, forest fires, volcanic emissions and vegetation (Cempel and Nikel, 2006). On contrary, the anthropogenic activities are the major contributor in sources of nickel which resulted from industrial and commercial uses. Generally Ni is found at low levels in the environment (US EPA, 2000). In landfill, Ni may have come from leaching of metals, electronic items, batteries and other waste type (Li *et al*, 2009b). Furthermore, it may result in atmospheric accumulation of nickel from combustion of coal, diesel oil and fuel oil, and the incineration of waste. Nickel is an important metal, heavily utilized in industry mainly due to its anticorrosion properties. Humans use nickel for many different applications. The most common application of nickel is the use as an ingredient of steal and other metal products. It can be found in common metal products such as jewelry. As a consequence, nickel-containing wastes such as spent batteries and catalysts are generated in various processes (Iyaka *et al.*, 2011).

Foodstuffs naturally contain small amounts of nickel while chocolate and fats have severely high quantities (Cempel and Nikel, 2006). Nickel uptake will boost when people eat large quantities of vegetables from polluted soils. Plants are known to accumulate nickel and as a result the nickel uptake from vegetables will be eminent (Andhale and Zimbare, 2012). Humans may be exposed to nickel by breathing air, drinking water, eating food or smoking cigarettes. Skin contact with nickel-contaminated soil or water may also result in nickel exposure. In small quantities nickel is essential, but when the uptake is too high it can be a danger to human health. An uptake of too large quantities of nickel has the following consequences, higher chances of development of lung cancer, nose cancer, larynx cancer and prostate cancer,

respiratory failure, birth defects, asthma and chronic bronchitis, allergic reactions such as skin rashes, mainly from jewelry, heart disorders and others (Rezaei *et al.*, 2011).

Nickel is released into the air by power plants and trash incinerators. It will then settle to the ground or fall down after reactions with raindrops. It usually takes a long time for nickel to be removed from air. Nickel can also end up in surface water when it is a part of the wastewater streams (Ntengwe and Maseka, 2006). The larger part of nickel compounds that are released to the environment will adsorb to sediment or soil particles and become immobile. In acidic ground, however, nickel is bound to become more mobile and it will often rinse out to the groundwater (Rulkens, 2005).

The high nickel concentrations on sandy soils can clearly damage plants and high nickel concentrations in surface waters and as a result, can diminish the growth rate of algae (Patil and Patel, 2012). Microorganisms can also suffer from growth decline due to the presence of nickel (Kuster *et al.*, 2006). For animals, nickel is an essential foodstuff in small amounts. But nickel is not only favorable as an essential element; it can also be dangerous when the maximum tolerable amounts are exceeded (Rai *et al.*, 2012). This can cause various kinds of cancer on different sites within the bodies of animals, mainly of those that live near refineries. However, nickel is not known to accumulate in plants or animals. As a result Nickel will not biomagnify up the food chain.

*e) Copper (Cu)*

Copper (Cu) is introduced into the environment through a number of natural methods and its origin in the waters is very diverse. The sources of copper in the environment are the extraction of copper from rock (rock weathering), minerals in soil, biological particles, including both living and dead organic material and volcanic action (Blossom, 2007). Waste or by-products produced as a result of human activities, either directly or

indirectly may leach into the water, deposited on the land and infiltrated into the ground may be presented in different chemical forms in accordance with its processes, which can sometimes reach a toxic level for soil organisms and plants. These releases can be originated from sewer overflows, water treatment plants or diffuse, for example, the water runoff washing of land, roads and roofs (Nirel and Pasquini, 2010). The most mention sources of copper entering landfills are paint, blades, and bottles caps, insecticides, pharmaceuticals and cosmetics (Fekiacova and Pichat *et al.*, 2015).

According to Fraga (2005), copper can be found in many kinds of food, in drinking water and in air. The absorption of copper is necessary, because copper is a trace element that is essential to human health. Although humans can handle proportionally large concentrations of copper, too much copper can still cause eminent health problems (Pandey, 2013). Copper concentrations in air are usually quite low, that exposure to copper through breathing is negligible. But people that live near smelters that process copper ore into metal do experience this kind of exposure. People that live in houses that still have copper plumbing are exposed to higher levels of copper than most people, because copper is released into their drinking water through corrosion of pipes (Georgopoulos *et al.*, 2001).

Occupational exposure to copper often occurs. In the working environment, copper contagion can lead to a flu-like condition known as metal fever (Verghese *et al.*, 2016). This condition will pass after two days and is caused by over sensitivity (Sengupta, 2013). Long-term exposure to copper can cause irritation of the nose, mouth and eyes and it causes headaches, stomach-aches, dizziness, vomiting and diarrhea (Dalapati *et al.*, 2011). Intentionally high uptakes of copper may cause liver and kidney damage and even death (Gaetke and Chow 2003). Whether copper is carcinogenic has yet been determined (Obiri *et al.*, 2010).

Study done by Osredkar (2012) indicates a link between long-term exposure to high concentrations of copper and a decline in intelligence among young adolescents. Industrial exposure to copper fumes, dusts, or mists may result in metal fume fever with atrophic changes in nasal mucous membranes (Kumar and Singh, 2014). Chronic copper poisoning may result in Wilson's disease characterized by a hepatic cirrhosis, brain damage, demyelization, renal disease, and copper deposition in the cornea (Pfeiffer, 2007).

When copper ends up in soil it strongly attaches to organic matter and minerals. As a result it does not travel very far and hardly enters groundwater. In surface water, copper can travel great distances, either suspended on sludge particles or as free ions (Bentum *et al.*, 2011). Copper does not break down in the environment and because of that it can accumulate in plants and animals (Tchounwou *et al.*, 2012). On copper-rich soils only a limited number of plants have a chance of survival. That is why there is not much plant diversity near copper-disposing factories. Due to the effects upon plants copper is a serious threat to the productions of farmlands. Copper can seriously influence the proceedings of certain farmlands, depending upon the acidity of the soil and the presence of organic matter. Despite of this, copper-containing manures are still applied.

Copper can interrupt the activity in soils as it can negatively influence the activity of microorganisms and earthworms. The decomposition of organic matter may seriously slow down because of this. When the soils of farmland are polluted with copper, animals will absorb copper and damage to their health (Ako *et al.*, 2014).

### 2.3 Soil Pollution from Landfill

Landfilling is the most frequent waste disposal method worldwide (Spokas *et al.*, 2006). It is recognised as being an important option both now and in the near future, especially in low-income and middle-income countries. Malaysia is a South East Asia country where landfill is important and where the standard of waste management needs to be improved since waste generation continues to increase with the economy and population growth (Ismail and Manaf, 2013). According to Ihedioha *et al.* (2016) a steady increase in population and a corresponding increase in the rate of waste generation from industrial and human activities but without an efficient waste management system can pose risks to the environment and to public health.

In Malaysia, landfills are being filled up rapidly due to the current daily generation of approximately 33,000 tonnes of municipal solid waste (Ministry of Urban Wellbeing, Housing and Local Government, 2005). Thus, this situation creates a need to improve landfilling practices for example, an implementation of a more sustainable landfilling technology. However, due to financial constraints, most landfills in Malaysia is usually lack of environmental abatement measures, such as leachate collection systems and lining materials in comparison to sanitary landfill which have appropriate leachate treatment pond and gas collection system, as well as, other sustainable landfilling technology (Ismail and Manaf, 2013).

Any existing waste disposal management system is challenged by lack of appropriate management plan, institutional framework and financial resources (Leung *et al.*, 2008). Without proper waste and landfill management especially in non-sanitary landfill environmental may occur. Non-sanitary landfill which consist of unlined cell pose a risk to the environment where leachate containing heavy metal from the soil may infiltrate into the groundwater and consequently result in groundwater pollution (Al Raisi *et al.*, 2014). The way the wastes are handled, stored, collected and disposed can caused

contamination to the surrounding environment and pose risks to public health (Misra and Pandey 2005).

Landfills are reported to be one of the sources of soil pollution due of the production of leachate and its migration through waste (Tamer *et al.*, 2011). Long term deposition of waste in landfill coupled with weathering could lead to accumulation of heavy metal in soil which will result in pollution. Previous studies have showed that improper collection, segregation, and disposing practices of municipal solid waste can produce leachates that contain high concentrations of ammonium, organic matter, and heavy metals (Tatsi *et al.*, 2002). These leachates may lead to mobilization of organic and inorganic toxic matters into groundwater and soils, and pose potential threats to local ecosystem health (Liu *et al.*, 2013).

There is a growing concern, on the build-up of heavy metal in soil and groundwater. Different kinds of wastes such as electronic waste are responsible for the presence of heavy metals in the landfills. The recent increase of generation and disposal of waste such as food cans and scraps metal, dumping of household hazardous waste and electronic waste such as batteries and old computer raise the question about the quantity of metals in waste disposal sites and their fate in the environment. This is because such wastes mainly contain lead, cadmium, mercury, arsenic, copper, zinc and others (Shaibu *et al.*, 2015).

#### **2.4 Total Heavy Metals in Landfill**

The term total metals refer to the concentration of metal determined in an unfiltered sample after vigorous digestion, or the sum of the concentration of metals in the dissolved and suspended fractions (Lindeburg, 2015).

In a study done by Kanmani and Ghandhimati (2013), an assessment of total heavy metal was conducted using soil sample from two MSW landfills. The results showed

that Mn was the highest heavy metal found in all samples with the ranges between 420.7 – 1711.6 mg/kg followed by Pb that ranged from 44.09 - 178.84 mg/kg. Based on the results obtained, the presence of heavy metals in all samples indicates that there is appreciable contamination of the soil due to leachate migration. This showed that the migration and distribution of the contaminants species are still localized and not diffused to a wider area. Similarly, Karim *et al.* (2014) reported that Cd and Co were insignificant, in two open dumping sites in Bangladesh. Cr, Cu, Mn, Ni and Zn are present at higher concentration in Matuail than in Khulna. Only Zn was observed to showed higher concentration in Khulna dumping sites than Matuail (Karim *et al.*, 2014). Domestic wastes are mostly disposed on the dumping sites. On the other hand, resources from the MSW were recycled both at the secondary and final disposal sites. This is resulted as the main factor of the lower content of heavy metals in the wastes at both dumping sites examined (Karim *et al.*, 2014).

Furthermore, study conducted by Esakku *et al.* (2003) on heavy metal concentration of MSW from Perungudi dumping ground (PDG) showed that the concentration of As, Hg, and Cd were less as compared to other metals. The highest concentration was Zn with (284 mg/kg) followed by Cr, Cu, Pb, Ni, Cd, As and Hg in the dumping ground. This may be attributed to the dumping of Zn and Cr containing wastes. The results obtained in this study were then compared with the Indian standard which showed Hg, Cr, and Pb exceeded the limit. However, when compared with USEPA standard the metals are within the standard limit. In another study by Hoque and Haque (2015) the concentration of Fe was the highest in both sampling locations with 14564 mg/kg and 9830 mg/kg waste at Matuail and Aminbazar landfill sites respectively. Hoque and Haque (2014) concluded that the level of Fe, Cu, and Ni were found to remain beyond the Bangladesh standard. As a consequence, there might be high risk of surface and

groundwater contamination together with the risk of the heavy metals to enter the food chain.

Oygard *et al.* (2008) conducted an assessment of heavy metal concentration in sediments from MSW sanitary landfill in Norway. The study was observed that, the sediments contained high concentration of Zn, Cu, Pb and Cd than those of Ni and Cr. The results can be explained by Ni and Cr being present in large area and surface density whereas Zn, Cr, and Pb are present in smaller area and surface density found in leachate. Seeping of leachate in soil would cause heavy metal with smaller area and surface densities accelerate their deposition and pre-concentration in the soil.

## **2.5 Speciation and Mobilization of Heavy Metals in Landfill**

There is no doubt that speciation analysis offers a great challenge for analysts. The proper approach for the sequential extraction and application of appropriate analytical techniques and instruments can encourage wider use of speciation analysis in the laboratory. Elemental speciation information is crucial today because the toxicity and biological activity of many elements depend not only on their quantities, but also on their oxidation states and chemical forms (Chen & Ma, 2001). Thus, sequential extraction (SE) (Tessier *et al.*, 1979) can provide information about the identification of the main binding sites, the strength of metal binding to the particulates and the phase associations of trace elements in sediment. This will provide better understanding on the geochemical processes governing heavy metal mobilization and potential risks induced (Isen *et al.*, 2013). Among the sequential extraction schemes proposed to investigate the distribution of heavy metals in soil and sediment involve, the five-step extraction schemes were developed by Tessier *et al.* (1979) and Yuan *et al.* (2004). This procedure was used most widely and has been used in variety of matrices and successfully applied for the determination of heavy metal in soils including municipal and industrial solid

waste dumpsite (Egila *et al.*, 2014; Oviasogie & Ndiokwere, 2008; Oygard *et al.*, 2008; Yusuf, 2007; Xiaoli *et al.*, 2007).

### **2.5.1 Sequential Extraction Schemes**

Sequential extraction chemically leaches metals out of soil, sediment and sludge samples (Yang *et al.*, 2009). The purpose of sequential “selective” extraction is to mimic the release of the selective metals into solution under various environmental conditions. One commonly used sequential extraction procedure is designed to partition different trace metals based on their chemical nature. The sequential extraction process is typically accomplished in four (4) steps using; (1) acetic acid to extract all exchangeable, acid and water soluble metals (2) hydroxy ammonium chloride to extract all reducible metals (3) hydrogen peroxide to extract all oxidizable metals and (4) aqua regia to extract all remaining, non-silica bound metals. In each of the steps, calculated concentrations of chemicals and buffers are added and the sample is shaken on an end-over-end shaker. The leachate from each step is then digested and analyzed. This multi-step procedure assures that all the metals of concern are completely extracted from the sample.

The results from all the different steps are calculated and used to determine the accurate concentrations under different conditions. Factors such as pH of the acid used for adjustment, temperature and duration of extraction are the critical factors that control the concentration of metal extracted from the sample. Sequential extraction procedure for Cd, Co, Cr, Cu, Fe, Mn Ni, Pb and Zn has been extensively studied in both river sediments and marine sediments. Total metal concentration is used as a criterion to assess the potential effects of sediment contamination which implies all forms of metals have equal impact on the environment. Although the total concentration

of trace elements in soil gives some indication on the level of contamination, it provides no insight into element bioavailability or mobility.

Elements in soil are present in various physicochemical forms, which in turn influence its availability. Sequential chemical extraction techniques have been widely used to examine these physicochemical forms, and thus to better understand the processes that influence element availability (Khalil, 2012). In defining the desired partitioning of trace metals, care was taken to choose fractions likely to be affected by various environmental conditions. According to Tessier *et al.* (1979), heavy metals are associated with five fractions.

**(a) Exchangeable (F1)**

The exchangeable fraction involves weakly adsorbed metals retained on the solid surface by relatively weak electrostatic interaction and metals that can be released by ion-exchangeable processes (Fernandez *et al.*, 2004). Remobilization of metals can occur in this fraction due to adsorption-desorption reactions and the lowering of pH (Lee and Saunders, 2003). Exchangeable metals are a measure of those trace metals which are released most readily to the environment. Corresponding metals in the exchangeable fraction represent a small fraction of the total metal content in soil and can be replaced by neutral salts. This fraction generally accounted for less than 2% of the total metals present in soil (Filgueiras *et al.*, 2002). Exchangeable fraction is also known as non-specifically adsorbed fraction, it can be released by the action of cations such as K, Ca, Mg or (NH<sub>4</sub>) displacing metals which weakly bond electro-statistically onto organic or inorganic sites. The common reagents used for the extraction of metals in this fraction are MgCl<sub>2</sub> and sodium acetate (pH 5.4) by acetic acid. Reagents used for this purpose are electrolytes in aqueous solution, such as salts of strong acids and bases or salts of weak acids and bases at pH 7. Other reagents showing similar properties have

seldom been used, such as nitrate salts (to avoid complexation that is too strong) or calcium salts ( $\text{Ca}^{2+}$  being more effective than  $\text{Mg}^{2+}$  or  $\text{NH}_4^+$  in removing exchangeable ions). Results obtained with these reagents give good correlation with plant uptake (Qasim *et al.*, 2015).

Heavy metals in this fraction is held by electrostatic adsorption and thus easily released through sorption and desorption processes (Kumar *et al.*, 2011). Neutral salts for example, magnesium chloride and potassium nitrate at neutral pH of soils serve as ion displacing extractant to aid the release of metal ions attached by electrostatic attraction to negatively charged sites of soil particles (Yong *et al.*, 2012). Furthermore, they can be replaced by competing cations because metals in this fraction are non-specifically adsorbed and ion exchangeable. Metals in the exchangeable metal in soils and sediments is labile, highly toxic and the most bioavailable fraction (Wang *et al.*, 2010).

**(b) Carbonate or acid extractable (F2)**

Carbonate tends to be a major adsorbent for many metals when there is reduction of Fe-Mn oxides and organic matter in the aquatic system. The most popular use reagent for the extraction of trace metals from carbonates phases in soil and sediment is 1M sodium acetate adjusted to pH 5.0 with acetic acid (Gleyzes *et al.*, 2002). The carbonate fraction is a loosely bound phase and bound to changes with environmental factors such as pH (Filgueiras *et al.*, 2002). The time lag for the complete solubilization of carbonates depends on some factors such as the type and amount of the carbonate in the sample, and particle size of the solid (Kaplan *et al.*, 2009). Extraction of metals from carbonates phases enhances the leaching of metals specifically sorbed to organic and inorganic substrates. In general, this fraction is sensitive to pH changes, and the metal

release is achieved through dissolution of a fraction of the solid material at pH close to 5.0 (Peng *et al.*, 2009).

Metals bound to carbonate minerals are also bioavailable for gut environment of benthic organisms (Wang *et al.*, 2010). Acidified acetate is used as an extractant to release metals precipitated or co-precipitated as natural carbonates (Yong *et al.*, 2012). 1 M solution of HOAc-NaOAc adjusted to pH 5 usually dissolves carbonate minerals such as dolomite and calcite releasing the metals bound to them without dissolving organic matter, oxides and clay minerals (Yong *et al.*, 2012 and Kumar *et al.*, 2011). Further, Tokalioglu *et al.*, (2000) stated that carbonates of sediments containing significant concentration of heavy metals and concentrations have been observed to be pH sensitive.

**(c) *Fe-Mn oxide (F3)***

Fe and Mn oxides exist as nodules, concretions, cement between particles or as a coating on particles and are excellent trace element scavengers (Kabata-Pendias, 2010 and Ikem *et al.*, 2003) and play important role in the mobility and behavior of trace metals (Kumar *et al.*, 2011 and Wang *et al.*, 2010). The residual phase represents metals largely embedded in the crystal lattice of the soil fraction and should not be available for remobilization except under very harsh conditions (Yusuf, 2007). The carbonate fraction is influenced by pH. Fe-Mn oxides are excellent scavengers of trace metals and sorption by these oxides tend to control Cu, Mn and Zn solubility in soils.

**(d) *Oxidizable fraction or fraction bound to organic matter and sulphides (F4)***

In organic phase, metallic pollutant bound to this phase are assumed to stay in the soil for longer periods but may be immobilized by decomposition process (Giacalone *et al.*, 2005). Under oxidizing conditions, degradation of organic matter can lead to a

release of soluble trace metals bound to metallic pollutant. The extracts obtained during this step are metals bound to sulphides (Kebir *et al.*, 2014). The organic fraction released in the oxidisable step is considered not to be bioavailable due to the fact that it is thought to be associated with stable high molecular weight humic substances that release small amount of metals in a slow manner (Rodgers *et al.*, 2015). The most commonly used reagent for the extraction of metals in organic phases is hydrogen peroxide with ammonium acetate re-adsorption or precipitation of released metals (Favas *et al.*, 2011). Other reagents such as  $H_2O_2$  / ascorbic acid or  $HNO_3 + HCl$  have been used which can dissolve sulphides with enhanced selectivity, but on the other hand, silicates are attacked to some extent (Smichowski *et al.*, 2005).

Metals may bind to organic materials such as detritus, living organisms or coatings on mineral particles (Tokalioglu *et al.*, 2000). Therefore, organic matter and sulphides are important factors controlling the mobility and bioavailability of heavy metals (Wang *et al.*, 2010). For instance, sulphides are major solid phases controlling the concentration of dissolved heavy metals (Wang *et al.*, 2010). Aside from soil organic matter effect on controlling the mobility of heavy metals, soil organic matter greatly affects sorption of heavy metal because they contain functional groups that are capable of complexing metals (Thomas, 2015). Thomas (2015) further reported that although metal bounded to the organic matter are temporarily inaccessible, they can be solubilized by chemical oxidation.

(e) ***Residual fraction (F5)***

Residual phase serve as a useful tool in the assessment of the long-term potential risk of heavy metal or toxic metals entering the biosphere. Digestion in strong acid such as nitric acid, hydrochloric acid or acid mixture such as aqua regia that do not dissolve the silicate matrix have been commonly used to leach out the recalcitrant metals that are

bound to the sediment in the residual phase (Baran and Tarnawski, 2015). Residual phase give an estimate of the maximum amount of elements that are potentially mobilisable with the changes in environmental conditions (Cappuyns, 2012). Moreover, primary and secondary minerals containing metals in the crystalline lattice constitute the bulk of this fraction (Czaplicka and Buzek, 2011). Its destruction is achieved by the digestion with strong acids, such as HF, HClO<sub>4</sub>, HCl and HNO<sub>3</sub>. The amounts of associated metals are also associated by some authors as the difference between the total concentration and the sum of the fractions of the metals extracted during the previous extraction steps (Wali *et al.*, 2015).

Metals in this fraction are mainly from primary and secondary minerals which occlude or “close up” metals within their crystal structures (Tokalioglu *et al.*, 2000). In natural conditions, metals in this fraction are practically inaccessible for living organisms and are said to be immobile (Soliman, 2012). Due to the fact that the concentration of metals in the residual fraction is largely controlled by the mineralogy and extent of their weathering (in the form which are not soluble under experimental conditions), they are considered to be held within the mineral matrix (Soliman, 2012). Therefore, metals in residual fraction are less toxic because they are not readily bioavailable (Soliman, 2012 and Wang *et al.*, 2010). In addition, a high concentration of metals in this fraction is an indication that there is less pollution of an environment from anthropogenic sources (Yu *et al.*, 2012).

In general, the F1 and F2 fractions of metals are most readily available to the living environment. The F3 and F4 fractions of metals are available under extreme condition, while F5 fraction is not easily available for uptake by plants under natural conditions (Singh & Kalamdhad, 2013). Metal fractionation using sequential extraction for example in study done by Xiaoli *et al.*, (2007) gave a good indicator of metal distribution in soil. Demonstrating the adsorption of trace metals in Fraction 1

(exchangeable). It was reported that, Zn and Cr were the highest in the exchangeable fraction, indicating that Zn has a high potential of creating hazardous effects on the environment (Xiaoli *et al.*, 2007). On the other hand, only a small fraction of Pb and Ni was present in the exchangeable fraction, and Pb and Ni showed almost no content in the exchangeable fraction in all of the samples. In another study conducted by Oygard *et al.*, (2008) small amounts of the Cd, Zn and Cu present in the soil were extracted in fraction 1, but Ni was the only metal where a significant fraction was extracted. This outcome is consistent with the findings by Xiaoli *et al.* (2007).

Study by Egila *et al.* (2014) reported that in the study of Emir Palace dumpsites the carbonate fraction has the highest concentration of Zn with the value of 27.80 mg/kg. This could be as a result of waste containing zinc materials are deposited in the dumpsite. In contrary, Cd and Cr marked as the lowest in concentration, probably due to the absence of cadmium and chromium in the waste dump. On the other hand, Oviasogie & Ndiokwere (2008) conducted a study following modification of the sequential extraction procedure to determine the fractionation of the soil. The results showed that higher concentrations of Pb were detected in the carbonate fraction of the soil. The high level of Pb in the carbonate fraction implied that the carbonate fraction provided sites for binding of Pb (Mashal *et al.*, 2009).

Yusuf (2007), in his study on fraction of heavy metals in waste site soil had observed that Zn and Cu were largely associated with Fe-Mn oxides. Fe-Mn oxides are excellent scavengers of trace metals and sorption by these oxides tend to control Cu, Mn and Zn in soil. Zn and Cu were found to be strongly bound in this fraction because it have stability constant high enough to be concentrated in fraction 2. Oygard *et al.* (2008) determined that in all soil samples all heavy metals studied (Cd, Cu, Cr, Fe, Pb, Mn, Ni and Zn) were detected in large concentration in this fraction. These heavy metals might be presented as metal oxides, but it is more likely that the metals are adsorbed in or onto

the large amounts of iron oxide present in the sediments. In this case, this confirms the scavenging effect that precipitating iron has on heavy metals in landfill leachate and in aquatic systems in general (Oygard *et al.*, 2008).

Zakari *et al.* (2011) conducted a sequential extraction of Cu, Cd, Pd and Zn from soil around industrial waste dumpsite and reported that Cu is associated mainly with the organic matter in which a greater extraction percentage was obtained in the organic fraction of the sites. This is resulted as high affinity of Cu to organic matter. In consistence, this study is the outcome of an assessment of heavy metal availability and speciation in soil from MSW landfill in Marrakech city conducted by Kennou *et al.* (2015) who reported that, Cu and Pb are mostly bound to organic matter in the proportions of 96.12 % (Cu) and 84.38 % (Pb), respectively. In another study by Yusuf (2007), a sequential extraction was used to fractionate Cd, Cu, Zn and Pb from soils near waste site. It was reported that a large portion of Pb was associated with organic matter, thus a high fraction of Pb was found in organic fractions (Yusuf, 2007).

According to Oygard *et al.* (2008), significant amount of Cr, Cu, Ni and Pb were found in residual fractions and only low concentration of Fe, Zn, and Cd were present in the residual fraction. This might be happened due to incomplete extraction of these metals in Fraction 3 but most likely due to the metals being present associated with poorly soluble minerals (Oygard *et al.*, 2008). In a separate study by Xiaoli *et al.* (2007) on soil from Shanghai refuse landfill showed that, Ni, Cd and Pb were also found mainly in the residual fraction. Similarly, the result of a study conducted by Abdus-Salam *et al.* (2011) determined that the major heavy metal constitute in residual fraction was Pb in association with the geochemical fractions.

## 2.6 Heavy Metals Pollution Indicator

Landfills contain wastes that could produce heavy metal contamination to the surrounding environment especially the soil (Kanmani & Gandhimathi, 2013; Wuana & Okieimen, 2011). Generally, municipal waste landfills produce leachate that infects soil, surface and even the adjacent ground waters. Assessment of heavy metal pollution could be done by using different indicators. Commonly, in numerous previous studies on heavy metal pollution in soil, pollution load index (Demie & Degefa, 2015; Aydi, 2015), index of geoaccumulation (Ihedioha *et al.*, 2016; Salah *et al.*, 2012), enrichment factor, contamination factor (Hassaan *et al.*, 2016; Ololade, 2014), risk assessment code (Huang *et al.*, 2015; Liu *et al.*, 2013) and others were often used as indicators to assess the level of heavy metals and its risk to the environment and human health.

Akbari (2016) investigated the rate of soil pollution with Cd, Cr and Pb in landfill soil of Meshgin city, Iran using factors of contamination factor and index of geo accumulation. The result of contamination factor showed that in most of sampling points, Pb and Cr metals contamination factor were in the range between no pollution to moderate pollution class while Cd were in no pollution class.

Furthermore, Demie & Degefa (2015) work on the heavy metals pollution of soil around open landfill of Shashemane city, Ethiopia and its potential of the environment and local community revealed that based on their contamination factor, the heavy metals were in order as  $Cd > Cr > Co > Pb > Mn > Ni$ , since the sampling area was highly strongly polluted by Cd and Cr while less contaminated by Ni. It is concluded that the area of study considerably polluted and deteriorated in terms of its quality (Demie & Degefa, 2015).

In addition, Aydi (2015) described the contamination of Pb, Cu, Cr, Zn, Ni and Cd in soil of landfill of Bizerti, Tunisia which demonstrate low contamination level for Ni, Pb, Zn and Cu while moderate contamination levels for Cr and Cd. The Cf value for Cd

(3.81) and Cr (1.06) were found high in the samples studied indicating that the soil are highly polluted by this two heavy metals. The remaining Cf value for other heavy metal in the studied area follow the decreasing in order Ni (0.45) > Pb (0.39) > Zn (0.31) > Cu (0.34) which showed low in contamination (Aydi, 2015).

Besides that, Hassaan et al. (2016) assessed soil contamination for some selected potentially hazardous element (Zn, Pb, Cd, Sr, Ni, Mn and Cr) in two landfill sites in Iraq using contamination factor where all elements showed moderate contamination except Cd that showed considerable contamination factor. The study suggested that the activities of the landfilling cause the contamination.

On the other hand, the distribution of metal speciation associated with different geochemical fraction is a critical parameter to assess the potential mobility and bioavailability of heavy metals in sediments. Evaluating metal speciation can provide detailed information concerning the origin, mobilization, and importantly, the toxicity and risk of heavy metals. Therefore, it is of considerable interest to distinguish and quantify the forms of these elements to obtain a better estimate of the potential and environmental impact of contaminated sediments. Risk assessment code which consist of a five-level classification; no risk (<1%), low risk (1– 10%), medium risk (11–30%), high risk (31–50%), and very high risk (>50%) uses the exchangeable fraction of metals as the index, representing a portion of metals in sediment readily released to overlying water (Perin *et al.*, 1985) and provides a potential risk assessment of metals to aquatic organisms (Bacon & Davidson, 2008 and Jain et al., 2004).

Risk assessment code mainly applies the sum of exchangeable and carbonate bound fractions for assessing the availability of metals in sediments. It is widely known that the higher percentage of non-residual (F1 + F2 + F3) fractions the sediments contain, the easier it is for metals to be released and the more bioavailable they are. Metals in the acid-soluble fraction function as short-term ecological risk indicators, since they are the

metals with the weakest bonds in sediments (Smol, 2008). During the aqueous phase, they become more easily bioavailable. Risk Assessment Code (RAC) was used to assess the potential mobility and hazard of specific metals based on the percentages of exchangeable metals and bound-to-carbonate metals in the sediment (Liu *et al.*, 2009).

In a study by Wang *et al.* (2010), RAC established by Perin *et al.* (1985) was used to evaluate the risk of Cd, Cu and Pb in sediments of Jinzhou Bay where Cd was found to show high risk compared to Cu and Pb which indicate medium risk. On the other hand, RAC was applied in different type of sample which is sediment/soil from landfill. For example, Li *et al.* (2014) who had conducted study on landfill of electrolyte Mn residue had concluded that Mn showed to be at high risk as compared to other tested heavy metals. Similarly, Xiaoli *et al.* (2007) and Huang *et al.* (2011) in their study both have demonstrated the use of RAC to assess the risk of heavy metals in each study site.

Speciation of heavy metals in soil/sediment influences their mobilizations which correspond to its toxicity. This has been proved in several studies (Yang *et al.*, 2014a; Gonzales *et al.*, 2013 and Yu *et al.*, 2013) which have utilized RAC for the determination of potential risk of heavy metals in their study samples. These studies showed consistent outcome due to similar study samples in which Pb was found to exhibit lower risk compared to other tested heavy metals as it is found in high percentage in residual fraction (non-mobile).

## CHAPTER 3: MATERIALS AND METHOD

### 3.1 Jeram Sanitary Landfill

Jeram Sanitary Landfill is operated by World Wide Landfill Sdn Bhd, under a 25 year concession agreement with the Kuala Selangor state government. This landfill was built on a private land which was previously an oil palm plantation. This landfill is layered with marine clay which is suitable for agricultural purpose, but also offered additional protection for the landfill cell construction. The landfill operates 365 days per year and receives an average of 2500 tonnes of waste daily.

The domestic and commercial wastes received by this landfill were from the city councils of Shah Alam and Petaling Jaya, the municipal councils of Subang Jaya and Klang, the district council of Kuala Selangor, and private waste collectors. The landfill began its operations on 1st January 2007, covering an area of about 160 acres. It is designed with a capacity to hold 6 million tonnes of waste. Waste was disposed into cells. The cells are alternately used that at any given year, there are active cells and closed cells. Active cell means the working surface of a landfill upon which MSW is deposited before placement of daily cover (Ministry of Environment, British Columbia, 2016). While closed cell can be defined as landfill that has reached its permitted waste capacity and has been permanently capped and certified as closed by the appropriate state regulatory agency (Seth, 2015). Hence, this study was carried out in both cells to assess the soil contamination in order to make comparison in terms of heavy metal mobilization from each cell.

### 3.2 Samples Collection

Samples were collected from one active and one closed cell. Eight sampling points from each cell were selected and the coordinates of the point were recorded (Table 3.1) and the map of sampling points at Jeram Sanitary Landfill is shown in Figure 3.1.

**Table 3.1:** Sampling locations for both active and closed cells

Active Cell		Closed Cell	
Points	Coordinate	Points	Coordinate
P1	3°11'26.86'' N 101°21'53.42'' E	P1	3°11'12.50'' N 101°22'1.53'' E
P2	3°11'28.73'' N 101°21'53.72'' E	P2	3°11'12.07'' N 101°21'59.89'' E
P3	3°11'29.36'' N 101°21'54.24'' E	P3	3°11'11.86'' N 101°21'58.65'' E
P4	3°11'39.98'' N 101°21'54.96'' E	P4	3°11'11.99'' N 101°21'57.49'' E
P5	3°11'30.69'' N 101°21'5.62'' E	P5	3°11'11.07'' N 101°21'57.66'' E
P6	3°11'31.24'' N 101°21'56.31'' E	P6	3°11'10.96'' N 101°21'58.66'' E
P7	3°11'32.15'' N 101°21'57.09'' E	P7	3°11'11.14'' N 101°21'59.78'' E
P8	3°11'31.36'' N 101°21'58.43'' E	P8	3°11'11.33'' N 101°22'1.28'' E



**Figure 3.1:** Map of sampling points at Jeram Sanitary Landfill

The soil samples were collected in polyethylene bags, 30 cm deep from the surface (Figure 3.2) in accordance to 2014 ASTM E – 1197 Standard Guidelines for Conducting Terrestrial Soil-Core Microcosm Test. The samples were labeled and immediately brought back to the laboratory. Large objects including stones, pieces of brick, concrete and plant fragment were removed by passing the soil through 2mm sieve.



**Figure 3.2:** Soil collection from the closed cell

The samples were then air-dried overnight in a fume hood (Figure 3.3) prior to analysis to remove of hazardous volatile components. Soil samples were then ground and homogenized using pestle and mortar to smaller particle and fine. The soil samples were then sieved through severs (mesh size 100) and stored in polyethylene bags. The air-dried samples were then coned and quartered (approximately 20g) and two portions of 1g sub-samples were removed for each method for sequential extraction.



**Figure 3.3:** Air dried soil in fume hood

### **3.3 Inductive Coupled Plasma-Mass Spectrometry Analysis**

An Inductive Coupled Plasma-Mass Spectrometry (ICP-MS, Agilent 7500a Series c) equipped with Babington nebulizer was used to determine the heavy metal concentration of Ni, Zn, Mn, Cu and Pb in soil samples. A glass double-path spray chamber and a standard quartz torch were operated at conditions as listed in Table 3.2. The operating parameters for working elements are set as recommended by the manufacturer. The solution was propelled into the nebulizer by a peristaltic pump. A centrifuge instrument, Kubota 2420 (speed range 0-6000 rpm, timer 0-60 min) were used to separate solid and liquid phases. A WTW 740 pH meter was used for pH reading of the solutions. A horizontal flask electrical shaker (220/60 Hz, Gallenkamp, England) was employed for the shaking of the samples at 3600 rpm. A water bath (96° c) and hot plate (90° c  $\pm$  5) was also used during sample digestion process.

**Table 3.2:** Instrumental parameters for trace element determination

Parameters	Conditions
RF power	1350 W
RF matching	1.6 V
Carrier gas flow rate	1.10 L/min
Peristaltic pump flow rate	0.1 rpm
Sample Uptake Time	30 sec
Sample Uptake Rate	0.4 r sec <sup>-1</sup>

### 3.4 Preparation of Solution

All reagents of analytical reagent grade and double deionized water (Milli-Q Millipore 18.2 M  $\Omega$ /cm resistivity) were used for all dilutions. All standards, reagents solutions and samples were kept in polyethylene containers. Acetic acid (glacial, 100% Fischer Scientific, Loughborough, Leicestershire, UK), hydroxylammonium chloride (ACROS Organics, NJ, USA), Hydrogen peroxide (30% Fischer Scientific, Loughborough, Leicestershire, UK) and ammonium acetate and HNO<sub>3</sub> (65% Suprapur Merck, Darmstadt, Germany) were from super pure quality. A multi-element standard solution IV for ICP-MS (Fluka, Switzerland) was used to prepare the series of standard solutions as listed below:

- i. Solution A (1 M of MgCl<sub>2</sub>) was prepared by dissolving 203.31g of MgCl<sub>2</sub> in 1000mL of deionized water and pH was adjusted by using NaOH.
- ii. Solution B (1 M of NaOAc) was prepared by dissolving 82.03g of NaOH in 1000mL of deionized water and pH was adjusted by 1 M of HOAc. 1 M of HOAc was prepared by dissolving 57.24 mL of HOAc in 1000 mL of deionized water.
- iii. Solution C (0.04 M of Hydroxyl ammonium hydrochloride (NH<sub>2</sub>OH-HCL)) in 25% HOAc was prepared by dissolving 0.28g of NH<sub>2</sub>OH-HCL in 250 mL of HOAc in 1000 mL of deionized water.

iv. Solution D (0.02 M of  $\text{HNO}_3$ ) was prepared by dissolving 1.4 mL of  $\text{HNO}_3$  in 1000 mL of deionized water.

v. Solution E (3.2 M of  $\text{NH}_4\text{OAc}$ ) was prepared by dissolving 246.66g of  $\text{NH}_4\text{OAc}$  in 200 mL of  $\text{HNO}_3$  and 800 mL of deionized water.

vi. Solution F (0.5 M of  $\text{HNO}_3$ ) was prepared by dissolving 35 mL of  $\text{HNO}_3$  in 1000 mL of deionized water.

### **3.5 Experimental Procedures**

#### **3.5.1 Pseudo total metal digestion**

Pseudo total metal concentrations of heavy metal were determined by aqua regia acid digestion method (Ivezić *et al.*, 2013). A total of 1g of dry soil sample was weighed to the nearest 0.01 g and transfer to a 50 mL tube. For the digestion, 10 mL of 1:1 Nitric Acid ( $\text{HNO}_3$ ) was added into the samples and the slurry were mixed and covered with a watch glass. The sample were then heated on the hot plate at  $90^\circ\text{C} \pm 5^\circ\text{C}$  and refluxed for 10 to 15 minutes without boiling. Then, the samples were allowed to cool in room temperature before the addition of 5 mL of concentrated  $\text{HNO}_3$ . The samples were further refluxed for 30 minutes. This step was repeated by adding of 5 mL of concentrated  $\text{HNO}_3$  until brown fumes emitted are no longer released, which indicates the complete oxidation reaction with  $\text{HNO}_3$ . The volume of the solutions were then reduced to approximately 5 mL and allowed to cool.

Next, 2 mL of deionized water and 3 mL of 30% Hydrogen Peroxide ( $\text{H}_2\text{O}_2$ ) were introduced into the samples. The container were covered with watching glass and returned to hot plate to start the peroxide reaction. The procedures employed were conducted cautiously to ensure that the losses do not occur due to excessively vigorous effervescence. The process was allowed to continue until effervescence present subsides and the samples were then cooled. Suitable volumes of 30% of  $\text{H}_2\text{O}_2$  were added

continuously into the solution until the bubble subsides. The samples were then heated during acid-peroxide digestion until the volume was reduced to approximately 5 mL.

Lastly, 10 mL of concentrated Hydrochloric Acid (HCl) was added to the digested samples and the containers were covered with watching glass. The samples were then refluxed at  $90^{\circ}\text{C} \pm 5^{\circ}\text{C}$  for 10 to 15 minutes (Figure 3.4).



**Figure 3.4:** Samples refluxion

After cooling, the samples were filtered through Whatman No. 41 filter using PE funnel and collected into 100 mL polyethylene volumetric flasks. Deionized water were added to the mark as diluting the samples (Figure 3.5) and extracts were the transferred into polyethylene container. Lastly, the extractant were stored at  $4^{\circ}\text{C}$  which ready for analysis by ICP-MS.



Figure 3.5: Sample dilution

### 3.5.2 Sequential extraction

The sequential extraction procedure used in this study is based on Tessier *et al.* (1979) and Standard ISO 11466. The chemical extraction was carried out progressively on sieved samples with initial weight of 1.0 g. The extraction and procedures were detailed as follows:

**a) Fraction 1 (Exchangeable)**

1.0 g of soil samples were extracted with 10 ml of Solution A [1M Magnesium Chloride ( $\text{MgCl}_2$ )] at pH 7 for 2 hours in room temperature with continuous agitation.

**b) Fraction 2 (weakly complexed and bound to carbonate)**

The residues from Fraction 1 were continuously agitated with 10 ml of Solution B [1M Sodium Acetate ( $\text{NaOAc}$ )] which already adjusted to pH 5 with Acetic Acid ( $\text{HOAc}$ ) at room temperature for 5 hours.

**c) Fraction 3 (Bound to Fe-Mn oxides)**

Residues from Fraction 2 were mixed with Solution C [20 mL of 0.04M Hydroxyl ammonium hydrochloride (NH<sub>2</sub>OH-HCl)] in 25% (v/v) of HOAc for 6 hours at 96 °C with occasional agitated. After the samples were allowed to cool, 20 mL deionized water were added.

**d) Fraction 4 (Bound to organic matter and sulphide)**

Residue from Fraction 3 was extracted with 3 mL of Solution D [0.02M HNO<sub>3</sub> and 5 mL of 30% H<sub>2</sub>O<sub>2</sub> (adjusted to pH 2 with HNO<sub>3</sub>)]. It was then heated to 85 °C for 2 hours. Then, 3 mL aliquot of 30% H<sub>2</sub>O<sub>2</sub> (adjusted to pH 2 with HNO<sub>3</sub>) was added into the samples at 85°C for 3 hours with intermittent agitation. After the samples were allowed to cool, 5 mL of Solution E (3.2 M NH<sub>4</sub>OAc in 20% (v/v) HNO<sub>3</sub>) were added, with continuous agitation for 30 min.

**e) Fraction 5 (Residual)**

The residues from Fraction 4 were digested with 9 ml of 12 M HCl followed by 3 ml of drop by drop 15.8 M HNO<sub>3</sub> to reduce foaming. After that, 5 mL of Solution F (0.5 M HNO<sub>3</sub>) were added and kept for 16 hours at room temperature to oxidize the organic matter in the soil. The mixture then were heated and maintained for 2 hours before cooled.

The samples were centrifuged at 3500 rpm for 8 min at room temperature for each extraction and the supernatants from each extraction were subjected to ICP-MS analysis. Prior to the initiation of the next extraction step, 10 mL deionised water was used to wash the samples and then the washed solution was discarded after centrifuged. All the experiments were carried out in triplicates to reduce the systematic error.

### 3.6 Heavy Metals Pollution Indicator

In order to assess the degree of heavy metals contamination in soil samples from active and closed cell of Jeram sanitary landfill, individual contamination factors ( $C_f$ ) of heavy metals and risk assessment code (RAC) were applied in this study. The results obtained indicate the potential implication of heavy metals found in soil to the environment.

#### 3.6.1 Contamination factor ( $C_f$ )

The determination of heavy-metal contamination factor is an important aspect that indicates the degree of heavy metals risk to the environment in relation with its retention time. A high contamination factor of heavy metals shows low retention times and high risk to the environment. The individual contamination factor ( $C_f$ ) of heavy metals was used to estimate the relative retention time of heavy metals in the soil. It is determined by dividing the sum of each heavy metal concentration in the mobile phase (non-residue phase) by its concentration in the residual phase (Ololade *et al.*, 2014; Salah *et al.*, 2012 & Nemat *et al.*, 2011). The level of soil contamination by heavy metals is expressed in terms of a contamination factor calculated as the following equation:

$$\text{Contamination factor (Cf) of each heavy metals} = \frac{F1 + F2 + F3 + F4}{F5}$$

The corresponding values were compared to the following classification and description by Hakanson, (1980) (Table 3.3).

**Table 3.3:** Contamination factor ( $C_f$ ) and level of contamination

Contamination factor	Contamination Level
$< 1$	Low contamination
$1 \geq C_f \geq 3$	Moderate contamination
$3 \geq C_f \geq 6$	Considerable contamination
$> 6$	Very high contamination

### 3.6.2 Risk Assessment Code (RAC)

Risk Assessment Code was applied to measure the risk of heavy metals in soil from landfill to the environment. The risk value was determined based on the percentage of the total metal content found in the first soil fraction by adding the percentage of F1 (exchangeable) and F2 (carbonate) (Yang *et al.*, 2014a). The values were compared to the following classification as described by Perin *et al.*, 1985 (Table 3.4).

**Table 3.4:** Classification of risk assessment code (RAC)

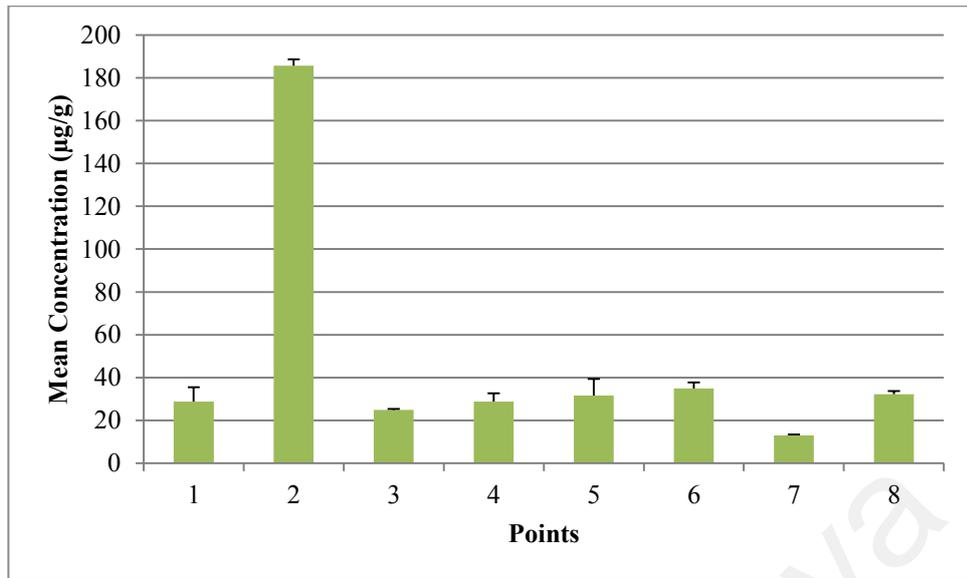
Criteria	Percentage (%)
No risk	$< 1$
Low risk	1 – 10
Medium risk	11-30
High risk	31-50
Very high risk	$> 50$

## CHAPTER 4: RESULTS AND DISCUSSION

### 4.1 Pseudo Total Heavy Metals Concentration

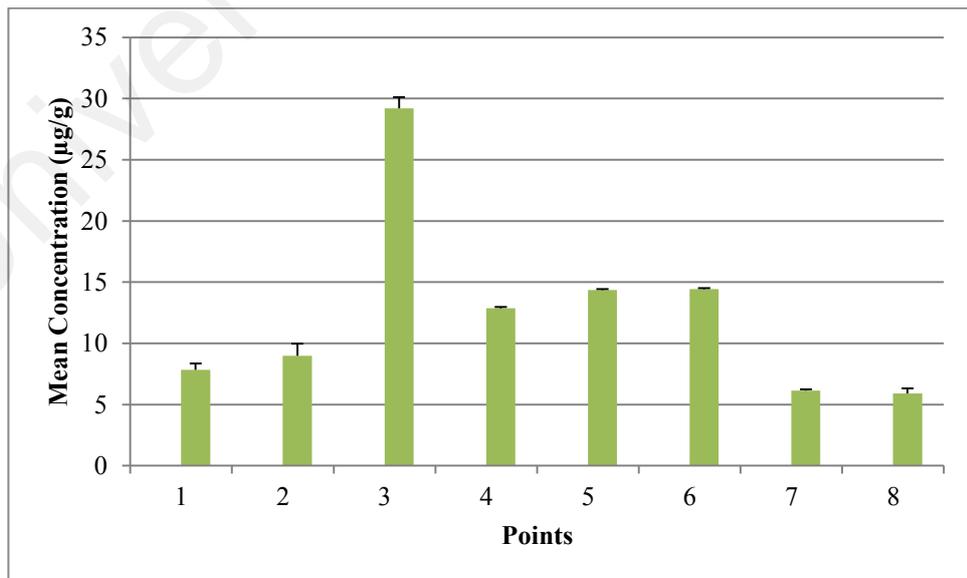
In this study, soil samples were collected from two different cells (active and closed cells) in Jeram Sanitary Landfill. Eight samples were collected from each cell. The following section highlights the result of this study.

Figure 4.1 and 4.2 show the total concentration of Pb in soil from active and closed cell, respectively. Pb was the highest in all samples from both cells. The highest concentration of Pb was found to be at active cell Point 2 with 185.6 µg/g. This is because, Point 2 was observed to have many discarded electronics items. Pb may possibly originated from electronic wastes as supported by Leung *et al.* (2008) who mentioned that, the main sources of Pb are from electronic wastes. This result is consistent with studies conducted by Li *et al.* (2009b) in which Pb was also recorded as the highest concentration among other heavy metal tested. Disposal of Pb-based batteries, paints and pipes seen at the site may possibly contribute to the increased of Pb concentration level in soil. This statement is supported by Al Raisi *et al.* (2014); Kanmani & Gandhimathi (2013) and Moturi *et al.* (2004) who stated that household hazardous wastes (e.g. batteries, paints, Pb contaminated toys and cleaners) discarded in landfill would cause Pb contamination in soil.



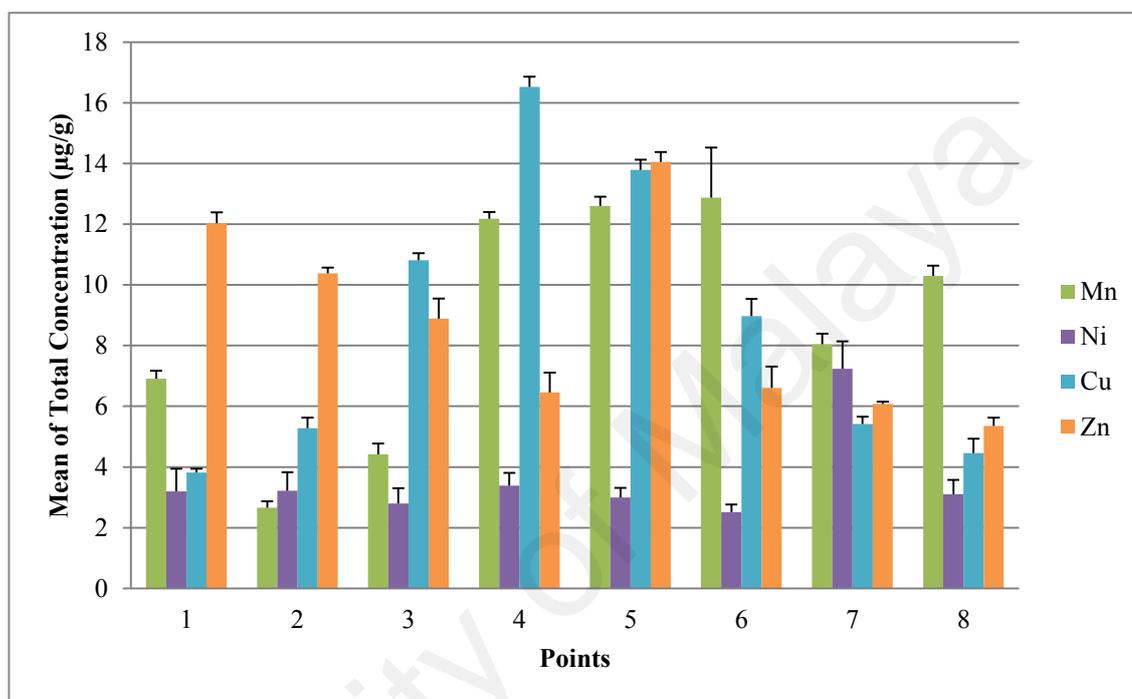
**Figure 4.1:** Concentration of Pb in active cell

On the contrary the concentration of Pb was found to be much lower in closed cell with the highest concentration at 29.21 µg/g. Pb in soil samples from closed cell was possibly from the disposal of waste prior to the closure of the cells. The concentration is much lower by 84.26 % because the closed cell has been inactive for more than five years.



**Figure 4.2:** Concentration of Pb in closed cell

Figure 4.3 and Figure 4.4 represent the concentration of Mn, Zn, Cu and Ni for each cell, respectively. Figure 4.3, shows that the active cell have high range concentration of Cu with 3.82 - 16.52  $\mu\text{g/g}$  followed by Zn with 5.35 - 14.05  $\mu\text{g/g}$ , Mn with 2.66 - 12.87  $\mu\text{g/g}$  and Ni 2.51 - 7.24  $\mu\text{g/g}$ .



**Figure 4.3:** Mean of Total concentration of Mn, Ni, Cu and Zn in active cell

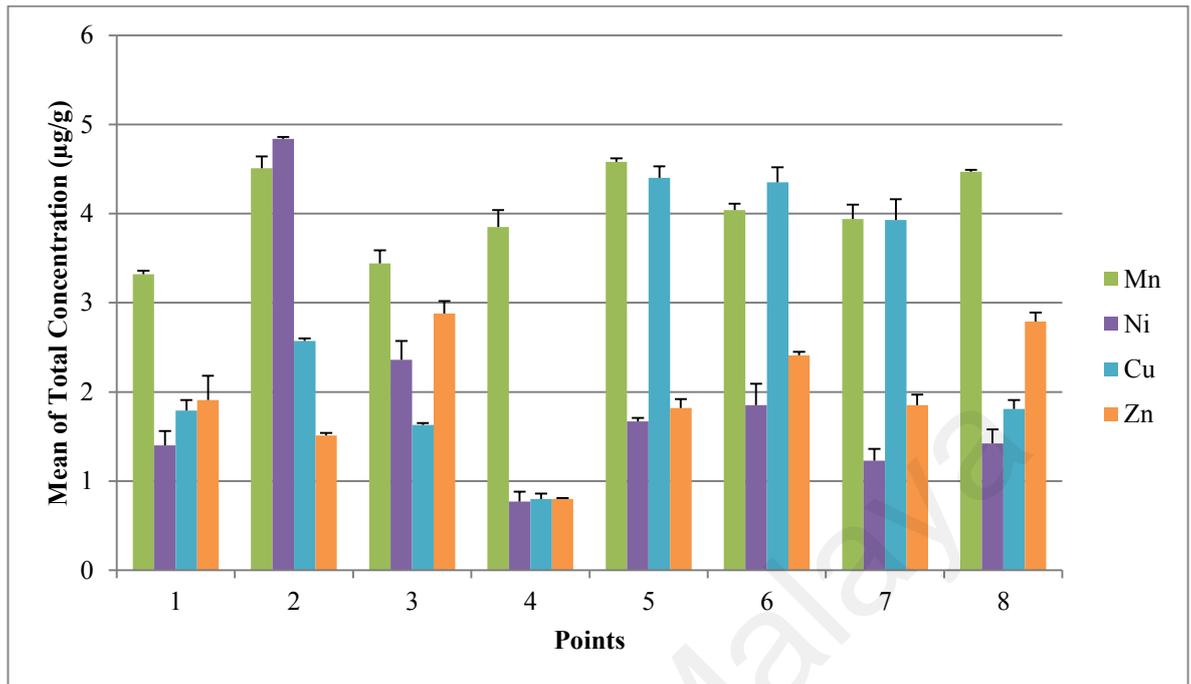
Cu has been reported to be in high concentration in most points. The high concentration of Cu may possibly originate from metals and electronic based waste. This agrees with the study done by Needhidasan *et al.* (2014) and Khaliq *et al.* (2014) which stated that most metal extracted by metals and electronic wastes is Cu. On the other hand, Ni was in low concentration in most samples from active cell with the lowest value of 2.51  $\mu\text{g/g}$ . This is due to the fact that in general Ni is found at low level in environment (US EPA, 2000). The traces of Ni detected in this study may have come from the leaching of metals, electronic items, batteries and other waste type similar to the observation reported by Li *et al.* (2009b).

As illustrated in Figure 4.4, closed cell has high range concentration of Mn with 3.32 – 4.58  $\mu\text{g/g}$  followed by Cu with 0.82 - 4.40  $\mu\text{g/g}$ , Zn with 0.80 - 2.88  $\mu\text{g/g}$  and Ni 0.77 – 4.84  $\mu\text{g/g}$ .

The soil sample from closed cell had high concentration of Mn which may probably resulting from the leaching of various waste disposed at site such as bottle caps, blades, cosmetics and others which can be seen along with garbage. According to Kanmani & Gandhimathi *et al.* (2013), a very high concentration of Mn and Cu was noticed in most area in the landfill they studied. It is concluded that sources of these metals are from paints, pigments, insecticides and pharmaceuticals. Therefore, this outcome supported the current findings. On the contrary, Ni has the lowest concentration of heavy metal in most sampling points except at point 2. It can be suggested that Ni was high at point 2 because it is not only link to the disposal of waste material but natural sources of Ni must be taken into consideration (Tangahu *et al.*, 2011). In addition, batteries containing nickel cadmium become the recycling option thus less disposal in the active cell (Bernardes *et al.*, 2004). Ni concentration at all sampling points in active cell were higher than closed cell except at point 2 of closed cell.

According to Filho and Miguel (2017), Cd and Ni are used in batteries and rechargeable batteries and are often discarded in household waste. Therefore, most likely the type of waste disposed at P2 containing batteries. Also, disposal of other electronic waste may contribute to the presence of Ni in soil as it contained Ni based printed circuit boards. Therefore, a higher Ni concentration found at P2.

In addition, According to Kjeldsen *et al.* (2002), sulfide is formed from sulfate reduction during waste decomposition in landfills. Sulfides and carbonates are capable of forming deposition of Cd, Ni, Zn, Cu, and Pb. Thus, decomposition processes causing in high concentration of Ni in closed cell.



**Figure 4.4:** Mean of Total concentration of Mn, Ni, Cu and Zn in closed cell

Table 4.1 and Table 4.2 show the comparison of total concentration for each heavy metal between active and closed cells correspond to each sampling points. From the data obtained, the mean concentrations of all heavy metals tested were computed. For active cell, it can be reported that the highest heavy metals concentration was Pb with 47.49 µg/g. On the other hand, for closed cell Pb also showed the highest concentration with 12.46 µg/g. Second highest concentration heavy metal in both cells is Mn followed by Zn, Cu and Ni.

Both soil samples collected from active and closed cells at Jeram Landfill were contaminated by Pb, Zn, Ni, Cu and Mg. However, as showed in the subsequent Table 4.1 and Table 4.2, it can be concluded that active cell showed higher concentration of heavy metals as compared to closed cell. This is because active cell are presently operating and continuously receiving disposed materials and wastes, that the heavy metals can be presumed to be coming from the disposed wastes. While, the heavy metals contamination in closed cell was originated mainly from previous disposal of

wastes. However, the landfill liner and final cover systems of the closed landfill makes the soil unexposed to the environmental conditions such as rain, sunlight and wind which prevent further chemical reaction taking place in soil. These results showed that the soil cover used in the closed cell managed to control the heavy metals from infiltrating into the soil (Natrah *et al.*, 2009).

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**Table 4.1:** Total Concentration of Heavy Metals (active cell)

Heavy metals	Total Concentration of Heavy Metals at Different Sampling Point ( $\mu\text{g/g}$ )								
	Point 1	Point 2	Point 3	Point 4	Point 5	Point 6	Point 7	Point 8	Mean
Mn	$6.91 \pm 0.26$	$2.66 \pm 0.21$	$4.42 \pm 0.35$	$12.18 \pm 0.22$	$12.60 \pm 0.31$	$12.87 \pm 1.66$	$8.04 \pm 0.35$	$10.3 \pm 0.33$	$8.75 \pm 0.46$
Ni	$3.20 \pm 0.74$	$3.22 \pm 0.61$	$2.80 \pm 0.50$	$3.38 \pm 0.43$	$3.00 \pm 0.31$	$2.51 \pm 0.25$	$7.24 \pm 0.9$	$3.10 \pm 0.47$	$3.56 \pm 0.53$
Cu	$3.82 \pm 0.12$	$5.20 \pm 0.35$	$10.80 \pm 0.23$	$16.52 \pm 0.34$	$13.79 \pm 0.34$	$8.97 \pm 0.57$	$5.42 \pm 0.24$	$4.46 \pm 0.47$	$8.63 \pm 0.33$
Zn	$12.03 \pm 0.36$	$10.38 \pm 0.19$	$8.89 \pm 0.66$	$6.46 \pm 0.65$	$14.05 \pm 0.33$	$6.6 \pm 0.71$	$6.08 \pm 0.07$	$5.35 \pm 0.28$	$8.73 \pm 0.41$
Pb	$28.80 \pm 6.72$	$185.60 \pm 3.00$	$24.94 \pm 0.49$	$28.86 \pm 3.78$	$31.58 \pm 7.75$	$34.86 \pm 2.83$	$13.07 \pm 0.45$	$32.3 \pm 1.57$	$47.49 \pm 2.46$

**Table 4.2:** Total Concentration of Heavy Metals (closed cell)

Heavy metals	Total Concentration of Heavy Metals at Different Sampling Point ( $\mu\text{g/g}$ )								
	Point 1	Point 2	Point 3	Point 4	Point 5	Point 6	Point 7	Point 8	Mean
Mn	$3.32 \pm 0.04$	$4.51 \pm 0.13$	$3.44 \pm 0.15$	$3.85 \pm 0.19$	$4.58 \pm 0.04$	$4.04 \pm 0.07$	$3.94 \pm 0.16$	$4.47 \pm 0.02$	$4.02 \pm 0.10$
Ni	$1.40 \pm 0.16$	$4.84 \pm 0.02$	$2.36 \pm 0.21$	$0.77 \pm 0.11$	$1.67 \pm 0.04$	$1.85 \pm 0.24$	$1.23 \pm 0.13$	$1.42 \pm 0.16$	$1.94 \pm 0.13$
Cu	$1.79 \pm 0.12$	$2.57 \pm 0.03$	$1.63 \pm 0.02$	$0.80 \pm 0.06$	$4.40 \pm 0.13$	$4.35 \pm 0.17$	$3.93 \pm 0.23$	$1.81 \pm 0.1$	$2.66 \pm 0.12$
Zn	$1.91 \pm 0.27$	$1.51 \pm 0.03$	$2.88 \pm 0.14$	$0.80 \pm 0.01$	$1.82 \pm 0.1$	$2.41 \pm 0.04$	$1.85 \pm 0.12$	$2.79 \pm 0.1$	$1.20 \pm 0.10$
Pb	$7.83 \pm 0.52$	$8.97 \pm 0.99$	$29.21 \pm 0.89$	$12.88 \pm 0.09$	$14.35 \pm 0.07$	$14.42 \pm 0.09$	$6.14 \pm 0.09$	$5.91 \pm 0.4$	$12.46 \pm 0.39$

Overall, for active cell, the sequence of heavy metals concentration in the soil sample is  $Pb > Mn > Zn > Cu > Ni$ . Meanwhile, the sequence of heavy metals concentration in the soil sample for closed cell is  $Pb > Mn > Cu > Ni > Zn$ .

#### 4.2 Speciation of Heavy Metals in Jeram Sanitary Landfill

Table 4.3 and 4.4 show the results obtained after the application of sequential extraction on all soil samples. Fraction 1 is the exchangeable fraction which is highly toxic and the most bioavailable fraction (Wang *et al.*, 2010). In this study, high concentration of Mn was found in most sampling points of active cell with 1.15  $\mu\text{g/g}$ . A high concentration of Mn was also found for closed cell with 1.21  $\mu\text{g/g}$ . A high concentration of Mn was extracted in this fraction because of the action of cations such as K, Ca, Mg, or  $\text{NH}_4$  in soil which displace the metals that is weakly bound to soils (Milivojević *et al.* 2011; Kaplan & Yaman (2009). On the other hand, the lowest concentration of heavy metal extracted in Fraction 1 was Pb with 0.03  $\mu\text{g/g}$  and 0.04  $\mu\text{g/g}$  in both cells, respectively. Yusuf (2007) also reported that, Pb was the least heavy metal extracted in exchangeable fraction because Pb is commonly associated with the oxidizable, Fe/Mn oxides (reducible forms) and carbonate species in the soil. In addition Jimoh and Sabo (2013) reported that, water soluble fractions of Pb were found in a very small percentage. This suggested that Pb in this soil may not be biologically available because water soluble fractions consist of metals species found in the soil solution.

Fraction 2 is the fraction in which metal bound to carbonate mineral. In Fraction 2 it can be seen that both Mn and Zn were mostly found in active cell with 11.11  $\mu\text{g/g}$  and 2.27  $\mu\text{g/g}$ , respectively. However, for closed cell only Mn was found at this fraction with 1.26  $\mu\text{g/g}$ . According to Billion *et al.* (2002) calcite in this phase has strong affinity for tested parameter especially Mn. Therefore, Mn was significantly extracted in

Fraction 2. Meanwhile, in this fraction, the lowest heavy metal concentration extracted for active cell was Cu with 0.07  $\mu\text{g/g}$  while for closed cell, it was Ni with 0.03  $\mu\text{g/g}$ . This outcome is similar to the finding by Jimoh & Sabo (2013) who reported that both Cu and Ni were less extracted in exchangeable and carbonate fractions. These results indicate a high Cu and Ni adsorption capacity by the soils affecting the concentration of Cu and Ni being extracted in this fraction (Nascimento *et al.*, 2003).

Fraction 3 is where metal bound to hydrous Fe-Mn oxide. Due to the large surface area, amorphous hydrous Fe-Mn oxide is one of the most important geochemical phases influencing the mobility and behaviour of traces metal (Violante *et al.*, 2010). In this fraction, Zn was the most extracted metal in both active and closed cell with 1.67  $\mu\text{g/g}$  and 2.67  $\mu\text{g/g}$ , respectively. This result is similar to findings from Jimoh & Sabo (2013) and Yusuf (2007) which reported high concentration of Zn in Fraction 3. This could be attributed to the high stability of Zn oxides in soils. On the other hand, Cu was found in low concentration for both cells at only 0.09  $\mu\text{g/g}$ . Cu were the least extracted element because Cu were mainly associated organic and residual fractions (Li *et al.*, 2000).

Fraction 4 is the fraction in which metal bound to organic matters and sulphides. Organic matter and sulphides are important factors that determine the mobility and bioavailability of heavy metal. In this fraction, in active cell a high concentration of Zn was found to bind to organic matter and sulphides with concentration of 2.04  $\mu\text{g/g}$ . The same result was obtained for closed cell in which Zn extracted the most with 1.01  $\mu\text{g/g}$ . Rozan *et al.* (2000) reported that Zn was the most extracted metal in Fraction 4. However, in most previous studies (Xiaoli *et al.*, 2007; Yusuf, 2007; Oviasogie and Ndiokwere, 2008; Wang *et al.*, 2010) Cu, Cd and Pb are the most commonly heavy metals associated in Fraction 4. The variation of these outcomes indicates that organic matter and sulphide are not the primary factor impacting the type of heavy metals to be extracted in Fraction 4 (Wang *et al.*, 2010). On the other hand, Pb was found to be the

lowest extracted heavy metal in both cells with 0.02  $\mu\text{g/g}$  and 0.05  $\mu\text{g/g}$ , respectively. Although, Pb is known to bind strongly to organic materials this current study show a low concentration of Pb was extracted in Fraction 4. As mentioned by Oygard *et al.* (2007), the level of organic matter in the soil does not necessarily indicate the types of metals to be extracted.

In Fraction 5 which is the residual fraction, metal are usually less toxic toward the environment. In this study, in both active and closed cells, Pb was mostly extracted in this fraction with 1.28  $\mu\text{g/g}$  and 1.29  $\mu\text{g/g}$  respectively. Egila *et al.* (2013), Abdus-Salam *et al.* (2011) and Oygard *et al.* (2007) have found a significant amount of Pb in this fraction. It was concluded in their study that the presence of Pb in this fraction might be due to incomplete extraction of Pb in Fraction 4 but most likely due to the metals being present associated with poorly soluble minerals. On the other hand, Ni was found to be less extracted compared to other metals with only 0.11  $\mu\text{g/g}$  in both cells because it is concentrated in exchangeable and oxidable fractions (Sepahvand and Forghani, 2012). In addition, according to Oygard *et al.* (2008) the low proportion of Ni in residual fraction is due to its conversion to carbonate in Fraction 2. Thus, it is highly extractable in Fraction 2.

**Table 4.3:** Concentration of metals extracted from each fraction of the Tessier sequential extraction (active cell)

Point	Fraction	Concentration of Heavy Metal ( $\mu\text{g/g}$ )				
		Mn	Ni	Cu	Zn	Pb
Point 1	Extraction 1	$0.73 \pm 0.05$	$0.09 \pm 0.01$	$0.10 \pm 0.00$	$0.12 \pm 0.01$	$0.17 \pm 0.04$
	Extraction 2	$6.89 \pm 0.20$	$0.08 \pm 0.004$	$0.08 \pm 0.00$	$0.32 \pm 0.01$	$0.14 \pm 0.03$
	Extraction 3	$1.66 \pm 0.03$	$0.12 \pm 0.003$	$0.10 \pm 0.00$	$1.10 \pm 0.02$	$0.74 \pm 0.01$
	Extraction 4	$0.68 \pm 0.01$	$0.18 \pm 0.003$	$0.37 \pm 0.02$	$0.73 \pm 0.01$	$0.05 \pm 0.01$
	Residue	$0.43 \pm 0.03$	$0.21 \pm 0.004$	$0.38 \pm 0.00$	$0.67 \pm 0.01$	$0.93 \pm 0.02$
Point 2	Extraction 1	$0.83 \pm 0.01$	$0.46 \pm 0.65$	$0.10 \pm 0.00$	$0.13 \pm 0.02$	$0.09 \pm 0.01$
	Extraction 2	$11.11 \pm 0.11$	$0.24 \pm 0.03$	$0.10 \pm 0.01$	$1.12 \pm 0.04$	$0.44 \pm 0.03$
	Extraction 3	$1.53 \pm 0.02$	$0.09 \pm 0.00$	$0.11 \pm 0.10$	$1.08 \pm 0.01$	$1.67 \pm 0.02$
	Extraction 4	$0.10 \pm 0.01$	$0.11 \pm 0.00$	$0.22 \pm 0.01$	$0.87 \pm 0.03$	$0.2 \pm 0.02$
	Residue	$1.07 \pm 0.03$	$0.18 \pm 0.01$	$0.80 \pm 0.02$	$0.62 \pm 0.03$	$1.22 \pm 0.01$
Point 3	Extraction 1	$0.86 \pm 0.04$	$0.59 \pm 0.10$	$0.41 \pm 0.01$	$0.72 \pm 0.03$	$7.30 \pm 0.22$
	Extraction 2	$0.15 \pm 0.02$	$0.11 \pm 0.00$	$0.07 \pm 0.01$	$0.19 \pm 0.03$	$0.14 \pm 0.02$
	Extraction 3	$0.22 \pm 0.01$	$0.02 \pm 0.00$	$0.09 \pm 0.00$	$0.21 \pm 0.01$	$0.39 \pm 0.01$
	Extraction 4	$0.96 \pm 0.00$	$0.40 \pm 0.02$	$0.22 \pm 0.00$	$1.72 \pm 0.03$	$0.04 \pm 0.00$
	Residue	$0.31 \pm 0.02$	$0.13 \pm 0.01$	$0.40 \pm 0.03$	$0.35 \pm 0.01$	$0.74 \pm 0.01$
Point 4	Extraction 1	$1.15 \pm 0.04$	$0.10 \pm 0.02$	$0.11 \pm 0.01$	$0.30 \pm 0.03$	$0.09 \pm 0.03$
	Extraction 2	$0.16 \pm 0.04$	$0.10 \pm 0.02$	$0.09 \pm 0.00$	$0.18 \pm 0.02$	$0.29 \pm 0.02$
	Extraction 3	$0.25 \pm 0.00$	$0.02 \pm 0.00$	$0.11 \pm 0.00$	$0.20 \pm 0.00$	$0.22 \pm 0.01$
	Extraction 4	$0.36 \pm 0.00$	$0.09 \pm 0.00$	$0.22 \pm 0.00$	$1.12 \pm 0.03$	$0.15 \pm 0.01$
	Residue	$0.42 \pm 0.02$	$0.11 \pm 0.00$	$1.02 \pm 0.02$	$0.22 \pm 0.01$	$0.88 \pm 0.03$
Point 5	Extraction 1	$0.92 \pm 0.10$	$0.10 \pm 0.01$	$0.09 \pm 0.00$	$0.17 \pm 0.02$	$0.08 \pm 0.04$
	Extraction 2	$0.07 \pm 0.01$	$0.10 \pm 0.02$	$0.08 \pm 0.00$	$0.16 \pm 0.02$	$0.20 \pm 0.00$
	Extraction 3	$0.11 \pm 0.00$	$0.13 \pm 0.01$	$0.09 \pm 0.03$	$0.41 \pm 0.01$	$0.14 \pm 0.01$
	Extraction 4	$0.79 \pm 0.01$	$0.68 \pm 0.01$	$0.93 \pm 0.00$	$2.04 \pm 0.01$	$0.50 \pm 0.00$

**Table 4.3:** Concentration of metals extracted from each fraction of the Tessier sequential extraction (active cell) (continued)

Concentration of Heavy Metal ( $\mu\text{g/g}$ )						
Points	Fraction	Mn	Ni	Cu	Zn	Pb
	Residue	$0.23 \pm 0.01$	$0.49 \pm 0.02$	$0.25 \pm 0.01$	$0.41 \pm 0.01$	$1.28 \pm 0.00$
Point 6	Extraction 1	$0.76 \pm 0.07$	$0.09 \pm 0.02$	$0.12 \pm 0.00$	$0.27 \pm 0.01$	$0.06 \pm 0.00$
	Extraction 2	$6.31 \pm 0.08$	$0.42 \pm 0.02$	$0.20 \pm 0.01$	$2.27 \pm 0.04$	$0.70 \pm 0.10$
	Extraction 3	$4.25 \pm 0.01$	$0.26 \pm 0.01$	$0.22 \pm 0.00$	$1.97 \pm 0.02$	$1.20 \pm 0.01$
	Extraction 4	$1.24 \pm 0.02$	$0.50 \pm 0.01$	$0.71 \pm 0.00$	$1.22 \pm 0.00$	$0.10 \pm 0.01$
	Residue	$0.97 \pm 0.00$	$0.32 \pm 0.02$	$0.33 \pm 0.01$	$0.27 \pm 0.00$	$0.92 \pm 0.01$
Point 7	Extraction 1	$0.59 \pm 0.03$	$0.08 \pm 0.00$	$0.08 \pm 0.00$	$0.12 \pm 0.01$	$0.04 \pm 0.01$
	Extraction 2	$0.17 \pm 0.01$	$0.08 \pm 0.01$	$0.08 \pm 0.00$	$0.22 \pm 0.03$	$0.17 \pm 0.01$
	Extraction 3	$0.19 \pm 0.01$	$0.12 \pm 0.00$	$0.09 \pm 0.01$	$0.63 \pm 0.01$	$0.15 \pm 0.01$
	Extraction 4	$0.40 \pm 0.02$	$0.23 \pm 0.01$	$1.15 \pm 0.01$	$0.94 \pm 0.00$	$0.02 \pm 0.00$
	Residue	$0.13 \pm 0.02$	$0.12 \pm 0.01$	$0.17 \pm 0.01$	$0.28 \pm 0.01$	$0.96 \pm 0.02$
Point 8	Extraction 1	$0.60 \pm 0.04$	$0.08 \pm 0.01$	$0.08 \pm 0.00$	$0.11 \pm 0.02$	$0.03 \pm 0.00$
	Extraction 2	$1.19 \pm 0.03$	$0.13 \pm 0.02$	$0.11 \pm 0.00$	$2.13 \pm 0.10$	$0.89 \pm 0.00$
	Extraction 3	$0.50 \pm 0.01$	$0.16 \pm 0.02$	$0.12 \pm 0.04$	$0.90 \pm 0.01$	$0.57 \pm 0.01$
	Extraction 4	$2.21 \pm 0.01$	$0.39 \pm 0.02$	$0.20 \pm 0.01$	$1.05 \pm 0.03$	$0.03 \pm 0.00$
	Residue	$0.35 \pm 0.03$	$0.11 \pm 0.00$	$0.35 \pm 0.02$	$0.34 \pm 0.01$	$0.71 \pm 0.01$

**Table 4.4:** Concentration of metals extracted from each fraction of the Tessier sequential extraction (closed cell)

Point	Fraction	Concentration of Heavy Metal ( $\mu\text{g/g}$ )				
		Mn	Ni	Cu	Zn	Pb
Point 1	Extraction 1	$0.97 \pm 0.05$	$0.11 \pm 0.01$	$0.08 \pm 0.01$	$0.26 \pm 0.02$	$0.06 \pm 0.03$
	Extraction 2	$0.49 \pm 0.06$	$0.03 \pm 0.00$	$0.07 \pm 0.00$	$0.04 \pm 0.01$	$0.11 \pm 0.02$
	Extraction 3	$0.37 \pm 0.01$	$0.22 \pm 0.02$	$0.12 \pm 0.01$	$1.28 \pm 0.02$	$1.19 \pm 0.11$
	Extraction 4	$0.60 \pm 0.01$	$0.15 \pm 0.00$	$0.27 \pm 0.01$	$0.63 \pm 0.00$	$0.14 \pm 2.02$
	Residue	$1.13 \pm 0.02$	$0.18 \pm 0.01$	$0.71 \pm 0.01$	$0.72 \pm 0.01$	$1.24 \pm 0.06$
Point 2	Extraction 1	$0.70 \pm 0.03$	$0.09 \pm 0.01$	$0.08 \pm 0.01$	$0.23 \pm 0.01$	$0.13 \pm 0.04$
	Extraction 2	$0.23 \pm 0.02$	$0.03 \pm 0.01$	$0.07 \pm 0.00$	$0.09 \pm 0.02$	$0.16 \pm 0.04$
	Extraction 3	$0.20 \pm 0.02$	$0.17 \pm 0.02$	$0.11 \pm 0.01$	$0.80 \pm 0.14$	$0.97 \pm 0.04$
	Extraction 4	$0.92 \pm 0.01$	$0.16 \pm 0.01$	$0.43 \pm 0.00$	$0.67 \pm 0.02$	$0.15 \pm 0.01$
	Residue	$0.61 \pm 0.01$	$0.15 \pm 0.00$	$0.40 \pm 0.01$	$0.42 \pm 0.01$	$0.88 \pm 0.00$
Point 3	Extraction 1	$0.57 \pm 0.01$	$0.07 \pm 0.00$	$0.07 \pm 0.00$	$0.28 \pm 0.02$	$0.08 \pm 0.01$
	Extraction 2	$1.26 \pm 0.01$	$0.06 \pm 0.01$	$0.08 \pm 0.00$	$0.25 \pm 0.05$	$0.11 \pm 0.03$
	Extraction 3	$1.08 \pm 0.10$	$0.27 \pm 0.04$	$0.10 \pm 0.01$	$2.02 \pm 0.03$	$0.61 \pm 0.04$
	Extraction 4	$0.12 \pm 0.00$	$0.18 \pm 0.01$	$0.15 \pm 0.01$	$0.67 \pm 0.02$	$0.13 \pm 0.00$
	Residue	$0.66 \pm 0.01$	$0.18 \pm 0.00$	$0.92 \pm 0.02$	$0.95 \pm 0.03$	$1.21 \pm 0.04$
Point 4	Extraction 1	$0.30 \pm 0.01$	$0.05 \pm 0.01$	$0.08 \pm 0.01$	$0.06 \pm 0.00$	$0.04 \pm 0.00$
	Extraction 2	$0.80 \pm 0.03$	$0.07 \pm 0.02$	$0.07 \pm 0.00$	$0.07 \pm 0.03$	$0.07 \pm 0.01$
	Extraction 3	$1.15 \pm 0.01$	$0.13 \pm 0.03$	$0.11 \pm 0.00$	$0.80 \pm 0.00$	$0.67 \pm 0.05$
	Extraction 4	$0.15 \pm 0.01$	$0.06 \pm 0.00$	$0.12 \pm 0.00$	$0.40 \pm 0.01$	$0.08 \pm 0.00$
	Residue	$0.70 \pm 0.10$	$0.12 \pm 0.00$	$0.22 \pm 0.02$	$0.31 \pm 0.01$	$0.72 \pm 0.03$
Point 5	Extraction 1	$1.21 \pm 0.02$	$0.08 \pm 0.01$	$0.07 \pm 0.00$	$0.44 \pm 0.06$	$0.04 \pm 0.01$
	Extraction 2	$0.83 \pm 0.04$	$0.07 \pm 0.00$	$0.10 \pm 0.03$	$0.55 \pm 0.01$	$0.23 \pm 0.03$
	Extraction 3	$0.64 \pm 0.06$	$0.31 \pm 0.01$	$0.13 \pm 0.01$	$2.67 \pm 0.42$	$0.91 \pm 0.12$
	Extraction 4	$0.10 \pm 0.01$	$0.07 \pm 0.01$	$0.12 \pm 0.00$	$0.73 \pm 0.01$	$0.02 \pm 0.01$

**Table 4.4:** Concentration of metals extracted from each fraction of the Tessier sequential extraction (closed cell) (continued)

Point	Fraction	Concentration of Heavy Metal ( $\mu\text{g/g}$ )				
		Mn	Ni	Cu	Zn	Pb
	Residue	$1.52 \pm 0.07$	$0.21 \pm 0.00$	$1.62 \pm 0.07$	$1.15 \pm 0.02$	$1.13 \pm 0.02$
Point 6	Extraction 1	$1.08 \pm 0.03$	$4.96 \pm 0.00$	$0.07 \pm 0.00$	$0.20 \pm 0.02$	$0.05 \pm 0.01$
	Extraction 2	$1.04 \pm 0.05$	$0.06 \pm 0.00$	$0.12 \pm 0.01$	$0.23 \pm 0.02$	$0.18 \pm 0.01$
	Extraction 3	$0.71 \pm 0.03$	$0.19 \pm 0.01$	$0.11 \pm 0.01$	$1.43 \pm 0.07$	$0.59 \pm 0.04$
	Extraction 4	$1.87 \pm 0.02$	$0.18 \pm 0.01$	$1.15 \pm 0.02$	$1.01 \pm 0.00$	$0.45 \pm 0.02$
	Residue	$1.13 \pm 0.03$	$0.20 \pm 0.01$	$0.94 \pm 0.03$	$1.08 \pm 0.00$	$1.15 \pm 0.01$
Point 7	Extraction 1	$0.65 \pm 0.01$	$0.06 \pm 0.00$	$0.08 \pm 0.00$	$0.20 \pm 0.03$	$0.09 \pm 0.01$
	Extraction 2	$0.97 \pm 0.05$	$0.05 \pm 0.00$	$0.17 \pm 0.02$	$0.23 \pm 0.04$	$0.26 \pm 0.01$
	Extraction 3	$0.33 \pm 0.02$	$0.18 \pm 0.01$	$0.14 \pm 0.01$	$1.04 \pm 0.12$	$0.89 \pm 0.10$
	Extraction 4	$0.09 \pm 0.01$	$0.05 \pm 0.01$	$0.11 \pm 0.00$	$0.41 \pm 0.02$	$0.05 \pm 0.00$
	Residue	$0.91 \pm 0.01$	$0.21 \pm 0.00$	$1.14 \pm 0.02$	$0.87 \pm 0.01$	$1.29 \pm 0.00$
Point 8	Extraction 1	$0.57 \pm 0.04$	$0.04 \pm 0.01$	$0.07 \pm 0.00$	$0.13 \pm 0.02$	$0.06 \pm 0.00$
	Extraction 2	$1.24 \pm 0.03$	$0.06 \pm 0.02$	$0.08 \pm 0.00$	$0.26 \pm 0.10$	$0.14 \pm 0.00$
	Extraction 3	$1.49 \pm 0.01$	$0.18 \pm 0.02$	$0.09 \pm 0.04$	$1.23 \pm 0.01$	$0.36 \pm 0.01$
	Extraction 4	$0.29 \pm 0.01$	$0.07 \pm 0.02$	$0.35 \pm 0.01$	$0.43 \pm 0.03$	$0.43 \pm 0.00$
	Residue	$0.62 \pm 0.03$	$0.11 \pm 0.00$	$0.54 \pm 0.02$	$0.49 \pm 0.01$	$1.12 \pm 0.01$

### 4.3 Potential Mobility of Heavy Metals in Jeram Sanitary Landfill

The mobility and immobility of heavy metals along with their availability in soil largely depend on their types of binding forms. Table 4.5 and Tables 4.6 shows the order of mobility (from most to least bioavailable) of heavy metals from F1 until F5 in both cells and each fraction is presented as a percentage of all fractions. The percentage of metals in each fraction was calculated as below;

$$\text{Percentage of each fraction} = \frac{\text{Concentration of heavy metal}}{\text{Total sequential extraction}} \times 100\%$$

Metals extracted from the F1, F2 and F3 are referred to the mobile fraction and the last two fractions (F4 and F5) are referred to as the non-mobile fractions.

In active cell (Table 4.5), Mn showed the greatest amounts in the bioavailable fractions, which ranged from 60 - 92 % extracted of the total contents in the F1, F2 and F3. The mobile fractions of Mn were higher than immobile fractions possibly from human-induced effects such as disposal of hazardous and industrial waste (Sungur *et al.*, 2016). As for closed cell (Table 4.6), Mn also recorded highest percentage in mobile fraction ranging from 13.9 - 28.2 %.

Meanwhile, for active cell the percentage of Cu in immobile fraction was higher than mobile fractions with 84 %. In closed cell, Cu was also found to be the most extracted in immobile fraction with compared to mobile fraction which only 14 %. It is noticeable that in both cell Cu showed higher percentages in the immobile fractions than mobile fractions. This finding is similar to Yusuf (2007) where Cu was largely associated with immobile fraction. A high percentage of Cu was found in carbonate and residual fraction in the landfill soil since Cu has a high stability state in the soil (Kabala and Singh, 2001 & Yusuf, 2007).

**Table 4.5:** Mobility of elements based on fractions for active cell

<b>Samples</b>	<b>Mobile % (F1+F2+F3)</b>	<b>Non-mobility % (F4+F5)</b>
P1	Mn (89.30) > Zn (52.38) > Pb (51.72) > Ni (42.65) > Cu (27.18)	Cu (72.82) , Ni (57.35)
P2	Mn (92.00) > Ni (73.14) > Zn (60.99) > Pb (60.77) > Cu (23.30)	Cu (76.69) , Pb (39.22)
P3	Pb (90.94) > Ni (57.60) > Mn (49.20) > Cu (47.80) > Zn (35.10)	Zn (64.89) , Cu (52.10)
P4	Mn (66.66) > Ni (52.38) > Pb (36.80) > Zn (33.60) > Cu (20.00)	Cu (80.00) , Zn (66.33)
P5	Mn (51.89) > Zn (23.19) > Ni (22.00) > Pb (19.09) > Cu (18.05)	Cu (81.00) , Pb (80.00)
P6	Mn (83.66) > Zn (75.20) > Pb (65.14) > Ni (48.42) > Cu (34.17)	Cu (65.82) , Ni (51.57)
P7	Mn (62.18) > Ni (44.44) > Zn (44.29) > Pb (26.87) > Cu (15.92)	Cu (84.08) , Pb (73.13)
P8	Zn (69.32) > Pb (66.82) > Mn (47.22) > Ni (41.86) > Cu (36.02)	Cu (63.95) , Ni (58.14)

**Table 4.6:** Mobility of elements based on fractions for closed cell

<b>Samples</b>	<b>Mobile % (F1+F2+F3)</b>	<b>Non-mobility % (F4+F5)</b>
P1	Zn (53.92) > Ni (52.17) > Mn (51.40) > Pb (49.63) > Cu (21.60)	Cu (78.40) , Pb (50.36)
P2	Pb (55.02) > Zn (50.67) > Ni (48.33) > Mn (42.48) > Cu (23.85)	Cu (76.15) , Mn (57.52)
P3	Ni (78.88) > Mn (78.86) > Zn (61.15) > Cu (18.93) > Pb (37.80)	Cu (81.06) , Pb (62.62)
P4	Mn (72.58) > Ni (58.14) > Zn (56.71) > Pb (49.37) > Cu (43.33)	Cu (56.67) , Pb (50.63)
P5	Zn (66.06) > Ni (62.16) > Mn (51.71) > Pb (50.64) > Cu (14.71)	Cu (85.29) , Pb (49.36)
P6	Ni (93.20) > Mn (48.54) > Zn (47.09) > Pb (33.88) > Cu (12.55)	Cu (87.45) , Pb (66.12)
P7	Mn (66.10) > Ni (52.73) > Zn (53.45) > Pb (48.06) > Cu (23.78)	Cu (76.22) , Pb (51.94)
P8	Mn (78.38) > Zn (63.78) > Ni (60.87) > Pb (26.54) > Cu (21.23)	Cu (78.76) , Pb (73.46)

### 4.3.1 Comparison of Potential Mobility of the Heavy Metals in Active and Closed Cells in Jeram Sanitary Landfill

In this study, comparison of the overall potential mobility of heavy metals observed in active cell and closed cell is shown in Figure 4.5. The potential mobility was calculated in terms of the average of metal percentages in mobile fraction (F1+ F2+F3).

$$\text{Potential Mobility} = \frac{\text{Sum of metal percentages in mobile fraction}}{8}$$

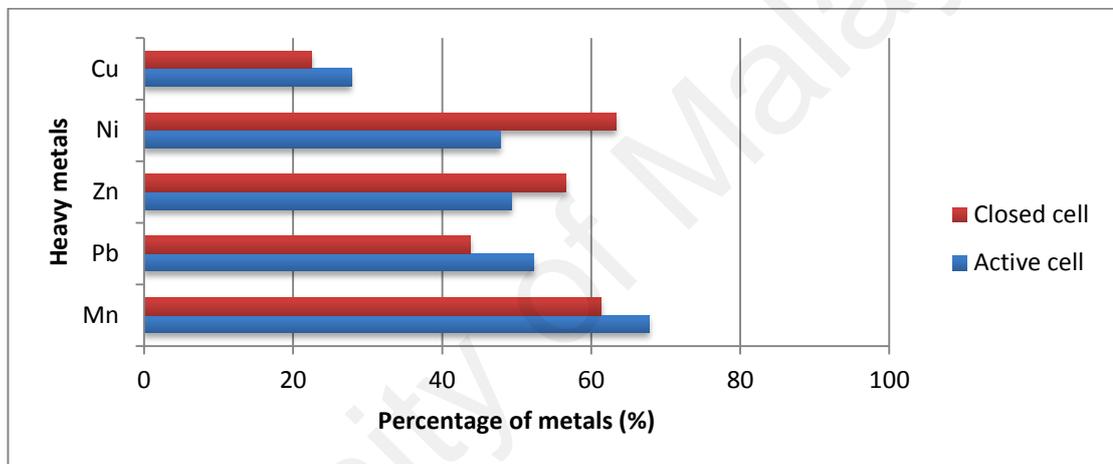


Figure 4.5: Potential mobility of heavy metals in active and closed cell in Jeram Sanitary Landfill

The dominant heavy metal in active cell was Mn (67.76%) followed by Pb (52.26%), Zn (49.26%), Ni (47.81%) and Cu (27.81%). Meanwhile, the dominant heavy metal in closed cell was Ni (63.31%) followed by Mn (61.26%), Zn (56.61%), Pb (43.82%) and Cu (22.5%). The overall mobility trend of heavy metals in each cell can be summarized as follow:

In active cell, Mn>Pb>Zn>Ni>Cu

In closed cell, Ni>Mn>Zn>Pb>Cu

The trend of mobility of heavy metals is different between active cell and closed cell. This could be due to the variation in the composition of waste materials received in both

cells. It was observed that in active cell, most of the wastes were steel-based waste in which could lead to the leaching of manganese into the soil. The finding is supported by Umm-khulthum *et al.* (2016) who stated that waste composition in soil affects the heavy metals mobility. Besides that, the difference of mobility trend between both cells could be due to the age and the status (inactivity) of the cell. According to Xiaoli *et al.* (2007), although the waste residue in closed landfill contained relatively high levels of heavy metals, it will only discharge a small quantity of trace heavy metals because the heavy metals have become stabilized overtime.

The implication of mobility of heavy metals to the environment is their potential for environmental toxicity due to bioavailability to flora and fauna. In active cell, the concentration of Pb was high. Based on the mobility trend, Pb was reported as mobile therefore it may present a threat to the environment. It can pose a threat towards the environment heavy metal that are in mobile fraction are most bio available, labile, can be easily release through sorption and desorption process and highly toxic. In closed cell, Pb concentration is also high but due to its immobility state, it may not present immediate threat to the environment. On the other hand, Mn was observed to be in mobile state in both cells. Therefore, based on this study, Mn is considered to have high mobility and bioavailability in the environment.

#### **4.4 Heavy Metals Pollution Indicator in Jeram Sanitary Landfill**

##### **4.4.1 Contamination Factor (Cf)**

The contamination factor (Cf) is widely used to evaluate the degree of heavy metal pollution in the soils. In this study, the Cf was computed for all soils samples using the concentration of the heavy metals. The calculated Cf for the studied heavy metals in soils of Jeram sanitary landfill is listed in Table 4.7 for active cell and Table 4.8 for closed cell.

Contamination factor for soil from active cell showed that the soil were in moderate to very high contamination class. The Cf value for Mn (12.29) and Zn (7.83) are relatively high indicating that the soils from active cell are highly polluted. The Cf value for heavy metals in the study follow the decreasing order of Ni (3.80) > Pb (2.74) > Cu (1.85) and demonstrated considerable contamination to moderate contamination.

**Table 4.7:** The value of contamination factor for soils from active cell

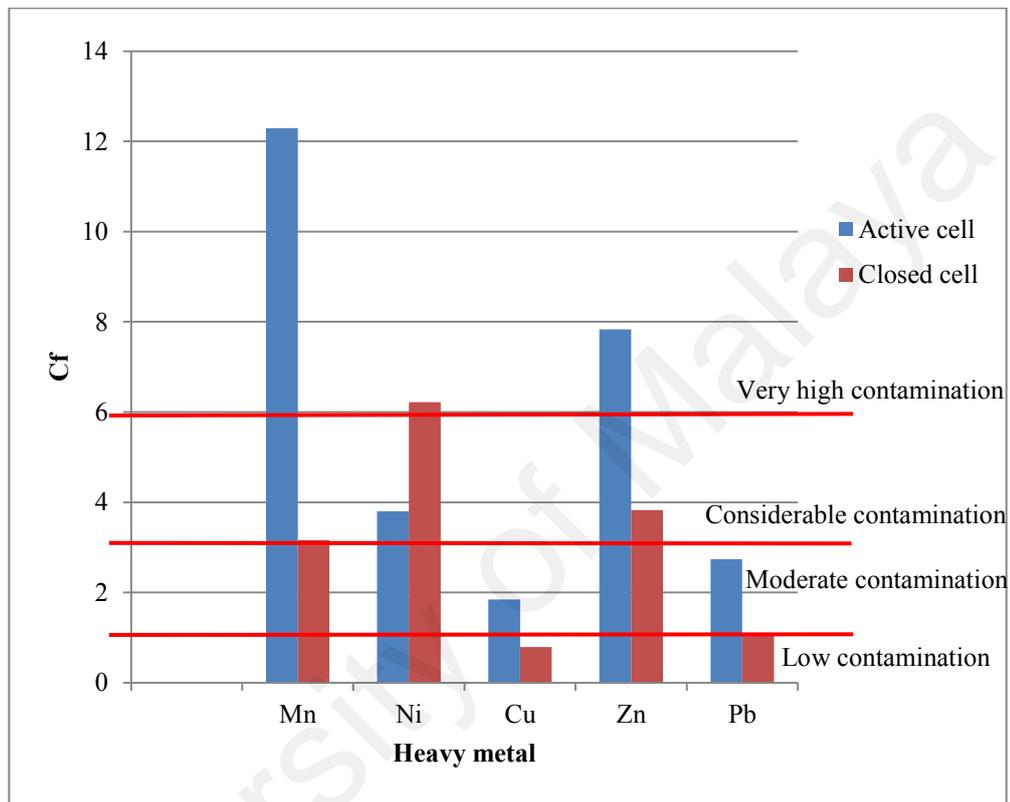
Heavy metals	Mean concentration					Contamination factor (Cf)	Classification
	F1	F2	F3	F4	F5		
Mn	6.43	26.05	8.71	6.74	3.91	12.29	Very high contamination
Ni	1.58	1.26	0.92	2.58	1.67	3.80	Considerable contamination
Cu	1.09	0.81	0.93	4.02	3.7	1.85	Moderate contamination
Zn	1.95	6.59	6.5	9.69	3.16	7.83	Very high contamination
Pb	7.81	2.97	5.08	5.08	7.64	2.74	Moderate contamination

In this study, the Cf for closed cell is represented in Table 4.8. Most of the heavy metals studied were in moderate contamination except for Ni which indicates very high contamination level. The trend is such that the highest Cf was observed for Ni while Cu showed low contamination value. In general, the increasing order of Cf value for all heavy metals is Cu (0.79) > Pb (1.08) > Mn (3.16) > Zn (3.83) > Ni (6.22).

**Table 4.8:** The value of contamination factor for soils from closed cell

Heavy metals	Mean concentration					Contamination factor (Cf)	Classification
	F1	F2	F3	F4	F5		
Mn	6.05	6.86	5.97	4.14	7.28	3.16	Moderate contamination
Ni	5.46	0.43	1.65	0.92	1.36	6.22	Very high contamination
Cu	0.6	0.91	0.91	2.7	6.49	0.79	Low contamination
Zn	1.8	4.95	11.27	4.95	5.99	3.83	Moderate contamination
Pb	0.55	1.26	6.19	1.45	8.74	1.08	Moderate contamination

Figure 4.6 demonstrates the comparison of heavy metals contamination between active and closed cells. It can be seen that all heavy metals recorded high Cf value in active cell than in closed cell except for Ni. Three heavy metals have recorded very high Cf; Mn, Zn and Ni.



**Figure 4.6:** Comparison of contamination factor values between active and closed cell

#### 4.4.2 Risk Assessment Code (RAC)

Risk Assessment Code was applied to measure the risk of heavy metals in soil from landfill to the environment. The risk assessment code for each heavy metal in all samples were determined based on the percentage of the total metal content that was found in the first soil fraction based on Tessier method. The values of RAC were obtained by adding the percentage of F1 and F2. The results obtained from RAC calculation indicate potential risk of heavy metals and the value were further compared to the following classification as described by Perin *et al.* (1985), >0% reflects no risk;

1 – 10% reflect low risk; 11-30% medium risk; 31-50% high risk; above 50%, the soil poses a very high risk and is considered dangerous, with metals easily able to enter the food chain.

Table 4.9 shows RAC value for each heavy metal in active cell. The results indicate a medium risk for most heavy metals with value between 11 – 30% except for Mn which was found to be at high risk in most samples. Based on previous sequential extraction results, Mn was found to be the most extracted heavy metal in Fraction 1 and 2 which indicate the most bioavailable metals. In addition with the RAC value, the percentage of Mn reflects very high risk (36.9 – 81.56 %).

**Table 4.9:** Comparison of RAC value for active cell

Samples	Parameters				
	Mn	Ni	Cu	Zn	Pb
P1	VH	M	M	M	M
P2	VH	VH	M	H	M
P3	H	VH	H	M	VH
P4	VH	H	M	M	M
P5	H	M	M	M	M
P6	VH	H	M	H	M
P7	VH	M	M	M	M
P8	H	M	M	H	H

L – Low risk; M – Medium risk; H – High risk; VH – Very high risk

On the contrary, the soil samples for the closed cell (Table 4.10) showed low risk for most heavy metal with RAC value 1 – 10 % except for also Mn which indicate medium to high risk with RAC value 9.71 – 41.01 %. On the other hand, Ni showed to

be very high risk at P6 with RAC value of 89.80%. These conditions may possibly pose risk to the environment as it is most labile, exchangeable and easily leach to the environment. This current finding is supported by previous studies done by Karim *et al.* (2014); Nda-Umar *et al.* (2012) and Prechthai *et al.* (2008).

**Table 4.10:** Comparison of RAC value for closed cell

Samples	Parameters				
	Mn	Ni	Cu	Zn	Pb
P1	H	M	M	M	L
P2	H	M	M	M	M
P3	M	L	L	L	L
P4	L	M	M	L	L
P5	M	M	L	L	L
P6	M	VH	L	L	L
P7	M	M	L	L	L
P8	M	L	L	L	L

L – Low risk; M – Medium risk; H – High risk; VH – Very high risk

## CHAPTER 5: CONCLUSIONS

The results obtained from the analysis indicated that the mean concentration of heavy metals in active cell were higher than that in closed cell. The mean concentration of Pb in active cell was higher (47.49  $\mu\text{g/g}$ ) than that in the closed cell (12.46  $\mu\text{g/g}$ ). Similar results were also obtained for Mn, Zn, Cu and Ni.

For the sequential extraction, Mn was detected in greatest amounts in mobile fractions in both active and closed cells. Meanwhile, Cu was at the highest concentration in immobile fractions in active and closed cell. On the other hand, the highest percentage of Pb in immobile fraction in active and closed cell was 80.0% and 50.36%, respectively. Thus, Cu and Pb showed the most non-mobile element in soils in both cells.

The potential mobility trend of heavy metals in Jeram Landfill can be concluded as; for active cell, the sequence is  $\text{Mn} > \text{Pb} > \text{Zn} > \text{Ni} > \text{Cu}$ . Meanwhile, the sequence for closed cell is  $\text{Ni} > \text{Mn} > \text{Zn} > \text{Pb} > \text{Cu}$ .

In this study, it was found that, the soil is possibly polluted particularly with Mn, Zn and Ni based on their contamination factor values. The high contamination factor of these heavy metals shows the increased possible risk to the environment. The results showed medium risk for active cell and low risk for closed cell for most heavy metals except for Mn which showed high risk in both cells. Overall, although Pb was found to be the highest concentration in all soil samples it is at low risk to the environment, due to its non-mobility state in soil.

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