CHAPTER 3

MATERIALS AND METHODS

3.1   The IPCC Waste Model

The IPCC Waste Model was developed by IPCC in 2006 for estimating CH_4 emission from landfills or any other solid waste disposal site (SWDs).

3.1.1 The IPCC formula(s)

The model follows the First Order Decay (FOD) method with assumption that the degradable organic component (degradable organic carbon, DOC) in the waste decayed slowly over the decade and formed CH_4 and CO_2. Therefore, the CH_4 emission from landfills for a single year was determined using Equation 1 as follows:

$$CH_4\ Emission = \left[\sum_X CH_4 generated_{X,T} - R_T \right] \cdot (1 - OX_T) \qquad \text{... Eqn. (1)}$$

Where:

CH_4 Emissions	=	CH_4 emitted in year T in Gg
T	=	Inventory year
X	=	waste category or type/material
R_T	=	recovered CH_4 in year T, Gg
OX_T	=	oxidation factor in year T, (fraction)

Only the fraction of CH_4 that is not recovered will be subjected to oxidation in the landfill cover, therefore the CH_4 recovered must be subtracted from the amount of CH_4 generated.

Decomposable degradable organic carbon (DDOC) is the part of the organic carbon that will degrade under the anaerobic conditions in landfill. The amount of CH_4 generated is calculated by multiplying the decomposable DDOC found in MSW with the fraction of CH_4 in LFG and CH_4/C molecular weight ratio (Equation 2).

$$CH_4 generated_T = DDOCm\ decomp_T \cdot F \cdot \frac{16}{12} \qquad\qquad \text{... Eqn. (2)}$$

Where:

CH_4 generated$_T$	=	amount of CH_4 generated from decomposables
DDOCm decomp$_T$	=	DDOCm decomposed in year T, Gg
F	=	fraction of CH_4 by volume in LFG
16/12	=	molecular weight ration CH_4/C (ratio)

The amount of decomposable material left in the landfill is calculated at the start of the year, T, with reference to the previous year, T-1. This means that the year in which the waste material was deposited in the SWDs is irrelevant to the amount of CH_4 generated each year. Equation 3 & 4 denotes the basics of this calculation.

$$DDOCm\ decomp_T = DDOCma_{T-1} \cdot (1 - e^{-k}) \qquad\qquad \text{... Eqn. (3)}$$

$$DDOCma_T = DDOCmd_T + \left(DDOCma_{T-1} \cdot e^{-k}\right) \qquad\qquad \text{... Eqn. (4)}$$

Where:

DDOCm decomp$_T$	=	DDOCm decomposed in year T, Gg

$DDOCma_T$	=	DDOCm accumulated at end of year (T), Gg
T	=	inventory year
$DDOCma_{T-1}$	=	DDOCm accumulated at end of year (T-1), Gg
$DDOCmd_T$	=	DDOCm deposited in year T, Gg
k	=	reaction constant or decay rate (y^{-1})
$t_{1/2}$	=	half-life time (y); $t_{1/2} = \ln(2)/k$

The index *m* used in all the above equations indicates mass in Gg. The DDOC is a product of amount of waste, fraction of DOC in waste, fraction of decomposable DOC in waste and part of waste that will decompose under aerobic condition in landfill before becoming an anaerobic condition which is interpreted as the methane correction factor (MCF). Equation 5 was used to calculate the DDOCm.

$$DDOCm = W \cdot DOC \cdot DOC_f \cdot MCF \qquad \qquad \text{... Eqn. (5)}$$

Where:

DDOCm	=	mass of decomposable DOC deposited, Gg
W	=	mass of waste deposited, Gg
DOC	=	degradable organic carbon, Gg C/Gg waste
DOC_f	=	fraction of decomposable DOC
MCF	=	CH_4 correction factor for aerobic decomposition

The five equations above were used to calculate CH_4 emission from JSL. The site-specific data for each parameter used within these equations were obtained from observations, previous records, calculations, as well as, from literature reviews. The key parameters used are discussed further in the following sections.

3.1.2 Key parameters of the model

3.1.2.1 Decay rate, k

The decay rate, k used in Equations 3 and 4, is dependent on many factors such as the composition of waste, half-life of the material or substance, climate condition of the landfill, characteristics of the landfill and waste disposal practices (Sormunen *et al*., 2013). The timing and rate of gas production also depends on how organic waste compounds degrade in landfills. The difficulty in determining and selecting the site specific value has been discussed by many researchers (Amini & Reinhart, 2011; Cruz & Barlaz, 2010; Tolaymat *et al*., 2010; Machado *et al*., 2009). The decay rate has a significant influence on the amount of LFG that can be produced in a landfill. In general, the higher the decay rate the higher the CH_4 production yield. **Table 3.1** shows the different *k* rates for different materials and also for the bulk waste based on IPCC model and several other literatures.

Table 3.1: Summary of decay rates, *k*, from different models and literature

Types of waste	IPCC (2006) estimates for Tropical Climate		Cruz & Barlaz (2010)	USEPA (2004)
	Default	Range		
Paper/textile waste	0.07	0.06 – 0.025	0.06[a]	0.10[c]
Wood / straw waste	0.035	0.03 – 0.05	0.03	0.03[d]
Other organic putrescible / garden and park	0.17	0.15 – 0.20	0.20[b]	0.33
Food waste / sewage sludge	0.4	0.17 – 0.7	0.14	0.29
Bulk waste	0.17	0.15 – 0.2	0.04	0.08[e]

a. weighted average of newsprint, coated paper, office paper
b. weighted average of branches, leaves, grass
c. weighted average of textiles, newspaper, office paper, magazines, corrugated cardboard
d. weighted average of grass, leaves, brush (Oshins & Block, 2000)
e. value used for landfill with high precipitation or leachate recirculation

Despite the above mentioned literature, the decay rate for a bulk waste approach can be calculated using the following formula (modified from USEPA, 2004) :

$$k = \left(3.2 \times 10^{-5} \times annual\ precipitation\ in\ mm\right) + 0.03 \qquad \text{... Eqn. (6)}$$

For JSL which records an estimated average of 2000mm rainfall per year the decay rate was estimated to be around 0.09 per year from Equation 6.

3.1.2.2 Degradable organic carbon (DOC)

Degradable organic carbon (DOC) from Equation 5 is the organic carbon fraction in waste that is subjected to biochemical decomposition or microbial degradation to produce LFG (IPCC, 2006). The organic part of each waste type is considered to have different decay rates (Thompson *et al*., 2009). Substances like cellulose and hemicellulose, commonly found in food and yard waste are readily degradable while lignin found in wood and newspapers is not readily degradable or might take a long period of time to degrade. **Table 3.2** depicts the DOC values and ranges used in IPCC waste model for a tropical climate.

Table 3.2: DOC values of various waste composition (IPCC, 2006)

Type of waste	IPCC default value	Range
Food waste	0.15	0.08 – 0.20
Garden	0.20	0.18 – 0.22
Paper	0.40	0.36 – 0.45
Wood & Straw	0.43	0.39 – 0.46
Textiles	0.24	0.20 – 0.40
Bulk Waste	0.18	0.12 – 0.28

In spite of the IPCC default value and range for DOC, a site-specific and more representative bulk waste DOC value can be calculated using the equation shown below:

$$DOC \ for \ JSL = (0.4 \times A) + (0.17 \times B) + (0.15 \times C) + (0.3 \times D) \ ... \ \textbf{Eqn. (7)}$$

Where A is the fraction of paper and textile waste, B is the fraction of garden and park waste, C is the fraction of food waste and D is the fraction of wood and straw waste. The decimal values in this formula refer to the fractional amount of DOC in each waste type (IPCC, 2006; Thompson *et al.*, 2009). All units are wet weight basis for kilogram carbon per kilogram waste. Therefore the IPCC DOC values and ranges, as well as, the site-specific calculation were used in this study.

3.1.2.3 Decomposable organic carbon fraction (DOC$_f$)

The DOC$_f$ from Eqn. 5 is the non-complete biodegradation of the organic carbon. The fraction of DOC which decomposed was based on a theoretical model where the variation depends on the temperature in the anaerobic zone of the landfill. The following formula is used to determine DOC$_f$ (IPCC, 2006):

$$DOC_f = 0.014 \cdot T + 0.28 \qquad\qquad ... \ \textbf{Eqn. (8)}$$

where T is the temperature of the anaerobic zone of the landfill. The value of DOCf ranges from 0.42 to 0.98 for a temperature range of 10˚C to 50˚C. However, the IPCC default value for DOC$_f$ is set at 0.5. It is known that temperature influenced the speed of the DOC converted to CH$_4$, however it is unclear to what extent the temperature in the

strictly anaerobic zone influenced this process (IPCC, 2006). In this study, the temperature used (in Eqn. 8) was 20˚C, 35˚C and 45˚C, as measured using a soil temperature probe in different locations of JSL. For the waste composition different values of DOC_f from literature was used.

3.1.2.4 Methane correction factor (MCF)

MCF in Eqn. 5 accounts for the factor that an unmanaged SWDs produced less CH_4 from a given amount of waste than an anaerobic site (IPCC, 2006). The MCF ranged from 0.4 – 1.0 depending on the landfill condition. **Table 3.3** shows MCF used for different classification of SWDs adapted from Jensen & Pipatti, (2006). For this study, MCF values of 0.5, 0.8 and 1.0 were evaluated.

Table 3.3: Values of methane correction factor (MCF) (Beh, 2012; IPCC, 2006)

MCF	Type of sites
1.0	Managed – anaerobic • A controlled placement of waste directed to a specific deposition areas • Control on scavengers and fire risks • Includes one of the following : Cover material / Mechanical compacting / leveling of waste
0.8	Unmanaged – deep (>5m waste) & high water content • Comprises SWDs which does not meet criteria for managed SWDs
0.6	Uncategorized SWDs • SWDs that do not meet the criteria for managed SWDs • However, has any of the following: cover material, leachate drainage system, gas collection system
0.5	Managed – semi anaerobic • Must have controlled placement of waste • Include the following structures: Permeable cover material, leachate drainage system, regulating pond, gas ventilation system
0.4	Unmanaged – shallow • SWDs that do not meet the criteria for managed SWDs • Has depth less than 5 meters

3.1.2.5 Fraction of CH_4 generated in LFG (F)

The fraction of CH_4 generated in LFG, F is used in **Eqn. 2.** In most landfills 50% of the LFG generated is CH_4. Therefore, the IPCC default value for F is 0.5 (Beh, 2012; IPCC, 2006). For this study, the F values of 0.5, 0.6 and 0.7 was used denoting to 50, 60 and 70% of CH_4 in LFG.

3.1.3   Site Description

The experimental site, Jeram Sanitary Landfill (JSL), is located in Jeram, Kuala Selangor in the state of Selangor, Malaysia. It is situated at 03˚11'20"N and 101˚21'50"E and has a total area of 160 acres. The landfill is operated by Worldwide Landfills Sdn Bhd since 2007, has a life span of 16 years and caters for 7 municipalities in the state of Selangor which has an approximate population of 5.6 million (JPM, 2013). It's design capacity is 1250 tonnes per day but currently receives around 2000 tonnes per day. It has a total estimated capacity of 8 million tonnes. The main type of waste received are 95% domestic wastes and 5% of other wastes. The landfill is covered with artificial liner (high density polyethylene – HDPE) and also has gas wells for gas capture. Currently, the LFG generated is flared as the volume is too low for power generation. Among the other facilities available in JSL are weighing bridge, gas vents, gas flaring units and leachate treatment pond. JSL is a part of the Integrated Waste Management System (IWMS) for the State of Selangor and Klang Valley, complementing other solid waste disposal projects in both regions, which have been identified by the Government of Malaysia.

Plate 3.1: Jeram Sanitary Landfill entrance

3.1.4   MSW data collection

MSW data was collected daily from JSL. The weighing bridge facility at site was used to weigh in and out the compactor lorries coming to the landfill. The lorries are weighed daily from 7am to 11pm. The mass of MSW deposited into the landfill is obtained by subtracting the mass of the compactor lorry before entering the landfill with the mass of the lorry exiting the landfill. The mass was then recorded in tonnes. The MSW data was collected from 2007 till 2013.

Plate 3.2: The weighing bridge facility at JSL

3.1.5 Manual sorting of waste

The sorting of MSW was carried out at the landfill site according to international standard ASTM D5231-92(2008)–*Standard Test Method for Determination of the Composition of Unprocessed Municipal Solid Waste* (ASTM, 2008). Mean composition of MSW was determined based on the collection and manual sorting of a number of samples of waste over a period of two weeks. As a good representation of the waste stream entering JSL, a compactor lorry was selected at random during each day of the sampling period. Each sorting sample weighed around 100 kg and was prepared properly (mixed, coned and quartered). After sampling, hand sorting was applied for the average classification of MSW onto the different categories. Each component category was then weighed and recorded.

3.1.6 Approaches to calculate methane from landfill

For the purpose of comparative studies, two approaches were used to calculate the CH_4 generated in JSL. In the first approach, the waste was summed up into a bulk waste category while in the second approach the different individual compositions within JSL were utilized. The two approaches are defined below.

3.1.6.1 The bulk waste approach

This approach is a single-phase model and was used to quantify the estimation for CH_4 generation from bulk waste deposited into the landfill. This option is often chosen for initial estimation of CH_4 from the landfills for inventory purposes. The parameters used were site-specific from JSL with suggested values from literature.

3.1.6.2 Waste composition approach

This approach is a multi-phase model and takes into account each type of degradable waste material (food, garden and park waste, paper and cardboard, wood, textiles, etc). In this approach the parameters are defined separately for waste material used depending on its characteristics.

3.1.7 Model Validation

The IPCC Waste Model was built to fit a wide range of data and circumstances. This model was validated using the actual measured data from JSL against the predicted value of the model. Error function analysis was used for the validation process.

3.1.7.1 Measured LFG from JSL

LFG was collected from the gas wells in JSL. The Memograph M Graphic Data Manager RSG40 by Endress and Hauser was used to determine the LFG flow rates. The memograph is a data logger which uses electronic data acquisition and variables such as pressure and temperature to calculate LFG flow rate in JSL. The measured CH_4 in percentage of volume over volume is the fraction of CH_4 in the LFG generated. LFG_{CH4} refers to the rate of CH_4 generated in an hour from the total LFG flow rate

3.1.7.2 Error Function Analysis

Error function analysis is a significant statistical analysis to prove the accuracy of model prediction. It was used to determine the deviation of the calculated values compared to the actual value. Three error functions: the fractional bias (FB), root mean square error (RMSE) and the normalized root mean square error (NRMSE) adapted from Cervone and Franzese, (2010) were used in this study and the equations are listed below:

$$Fractional\ bias\ (FB) = 2\left|\frac{\overline{c_m} - \overline{c_a}}{\overline{c_m} - \overline{c_a}}\right| \qquad \ldots \text{Eqn. (9)}$$

$$Root\ mean\ square\ error\ (RMSE) = \sqrt{\frac{\overline{(c_m - c_a)^2}}{\overline{c_m}^2}} \qquad \ldots \text{Eqn. (10)}$$

$$Normalized\ root\ mean\ square\ error\ (NRMSE) = \sqrt{\frac{\overline{(c_m - c_a)^2}}{\overline{c_m}\,\overline{c_a}}} \qquad \ldots \text{Eqn. (11)}$$

Where c_m and c_a are the measured CH$_4$ from JSL and calculated CH$_4$ from the model. Subsequently, model calibration was performed with the parameters showing the least error function from the **Equations 9 to 11**.

3.1.8 Modal calibration

The IPCC Waste model was calibrated using the results from error function analysis. The error estimates of more than 60% were reevaluated and new figures with errors below (30 ± 20) % were assigned for new set of proposed site specific values. These values were later used for calculating CH$_4$ generation in a temporarily closed cells in which where the field measurements were conducted.

3.2 Field measurements of LFG

Field measurements were carried at Phase 2 (**Plate 3.3**) waste cell in JSL. This cell was actively filled with MSW in 2008 and 2009 before its closure with a temporary cover of clay in 2010. These measurements were carried out for six months to determine the CH$_4$ flux emission from landfill surface. Before the measurements were done on a weekly basis, grid points were setup to estimate the area of analysis, as well as, for easy identification of measuring points for repeated measurements.
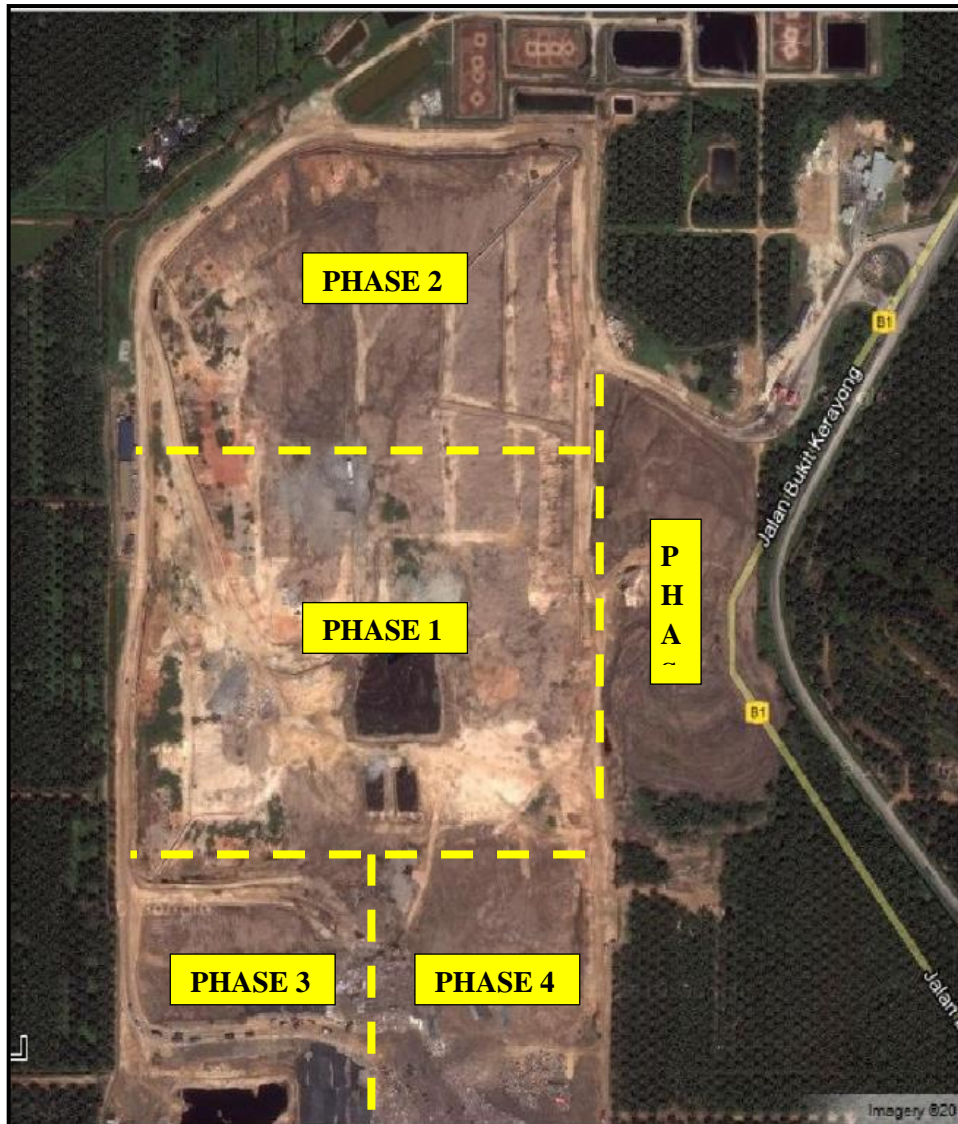
Plate 3.3: JSL aerial view (Worldwide, 2012)

3.2.1 Grid points setup at landfill

CH_4 emissions from the surface of the cell in terms of spatial variability were quantified during the field studies. An area of 22,500 m^2 of the cell surface was overlaid with square centers marking the grid that were studied. Each cross section of the grid was marked with wooden sticks. The distance between each stick was 50m. Measurements of flux were done in duplicates around the grid point to satisfy the

minimum criteria of sample size (n ≥ 20-30; n is the number of samples) for unbiased

statistical data (Statistics Handbook, 2009). The **Figure 3.1** and **Plate 3.4** show the grid

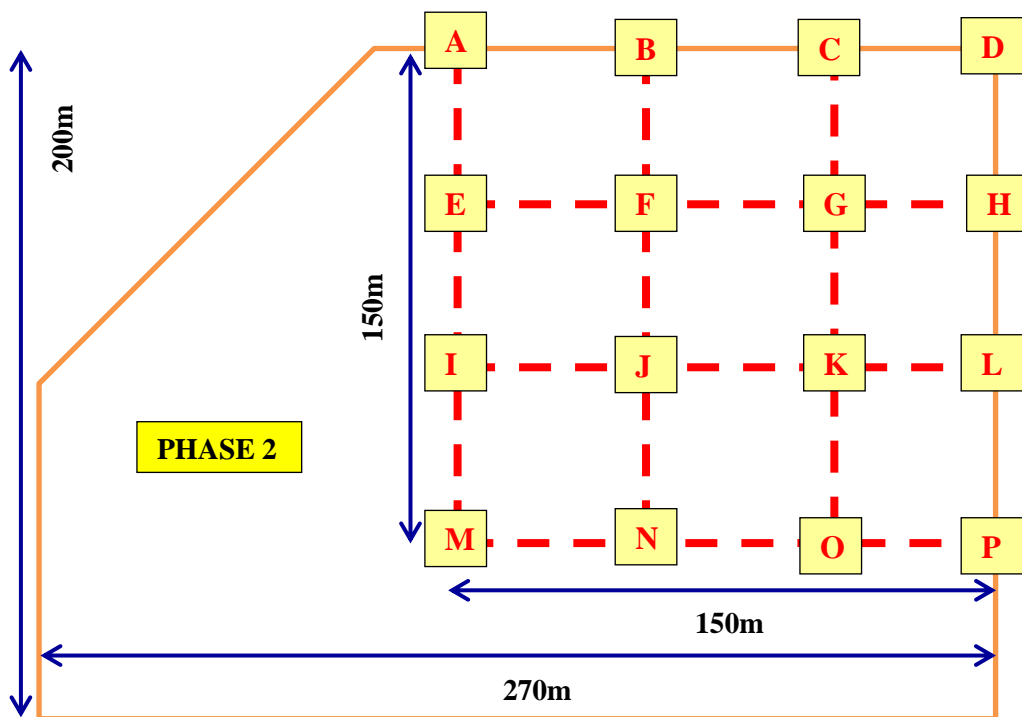overlay in JSL and the preparation for grid point setup, respectively.

Figure 3.1: Grid points overlay on Phase 2 at JSL

Plate 3.4: Wooden sticks used to mark the grid points at 50m distance

3.2.2 Design of flux chamber

The flux chamber dimension was designed according to volume to area (V/A) ratio found in literature, ranging from $60Lm^{-2}$ to $300Lm^{-2}$ (Fourie and Morris, 2004). For this study, a static flux chamber made from plexi glass and V/A ratio of $300Lm^{-2}$ was fabricated. The chamber has a square base of $625cm^2$ and height of 33cm. Figure 3.2 shows the schematic diagram of the flux chamber.
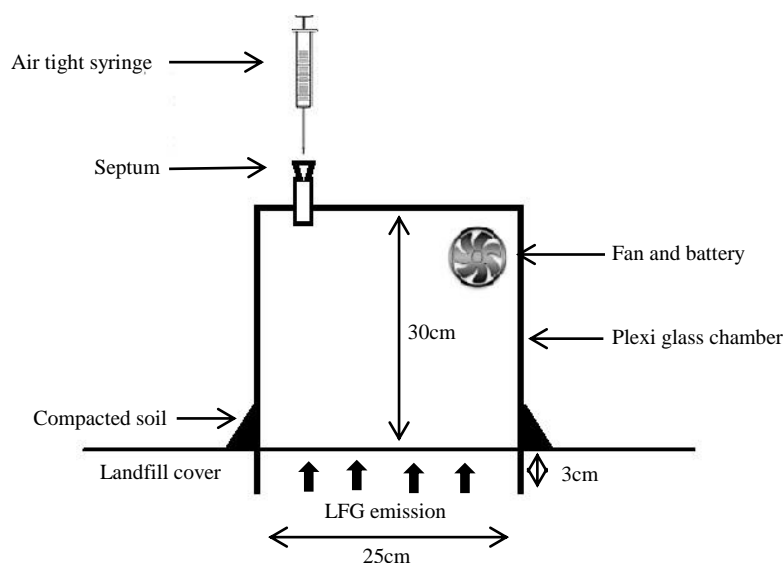
Figure 3.2: Static flux chamber

3.2.3 Method of flux sampling

The flux chamber was embedded into the landfill surface. It was sealed with compacted soil to avoid air intrusion and was placed for a duration of 30 minutes. A 10ml gas sample was drawn from the septum of the chamber using a 60ml air tight syringe at 10 minute intervals, as shown in **Plate 3.5**. Then, the gas was transferred to 10ml air tight vials and analyzed using a gas chromatograph (GC). Details on GC are explained in the following subtopic. This method was repeated weekly for a period of six months for the rainy months (October to December) and dry months (May to July) with duplicate sets of measurement each time.
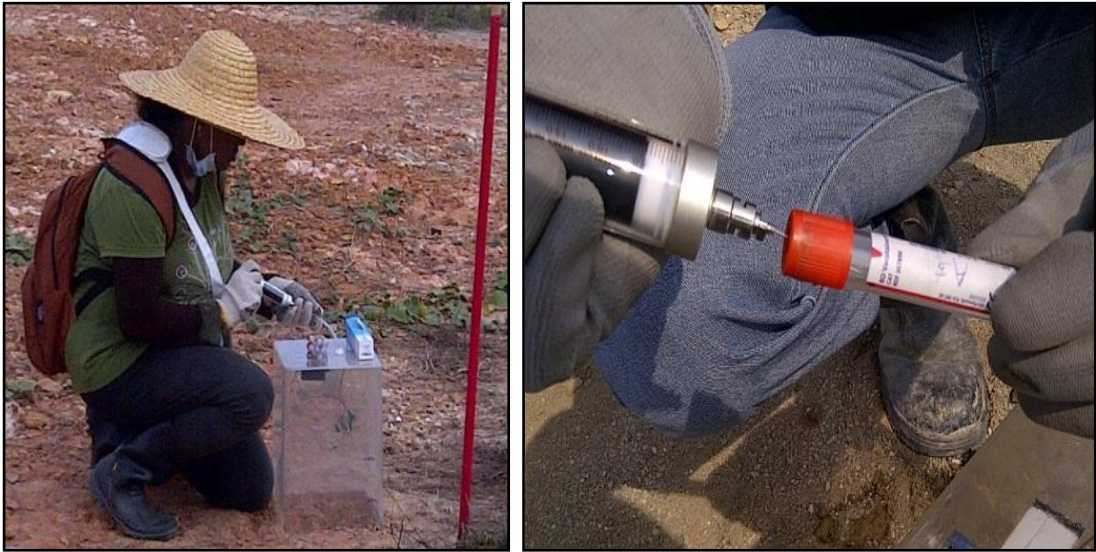
Plate 3.5: Gas extraction from flux chamber

3.2.4 Calculation of CH$_4$ flux

The basic equation for flux calculation is as below (Abushammala *et al*., 2012; Wangyao *et al*., 2010; Senevirathna *et al*., 2007; Spokas *et al*., 2006; Fourie & Morris, 2004)

$$\text{Flux,} = \left(\frac{V}{A}\right) \cdot \left(\frac{dC}{dt}\right) \cdot K, \qquad \text{… Eqn. (12)}$$

Where:

	=	CH$_4$ flux, g m^{-2} day^{-1}
V	=	volume of flux chamber, m^3
A	=	enclosed area by the chamber, m^2
$\frac{dC}{dt}$	=	rate of increase in gas concentration
K	=	correction factor

K is the correction factor for pressure, temperature and conversion of gas volume to mass based on ideal gas equations.

3.2.5 Flux contour analysis

The flux reading obtained from the formula was converted into contour plots for geospatial analysis. Two common interpolation methods were used for this analysis; kriging and inverse distance weighing (IDW) (Spokas *et al.*, 2003). Kriging the spatial dependency from semivariograms were used to predict flux reading at unknown location based on the sampling location (Mackie & Cooper, 2009). In the IDW technique the contours were interpolated using the inverse of the separation distance to a power of 2 (Abushammala *et al.*, 2012; Bella *et al.*, 2011; Wangyao *et al.*, 2010). The data generated by both the methods were later plotted into variograms (contour plots) to determine its spatial and temporal variability. The Surfer 10 (version 10) software for countering and 3D surface mapping by Golden Software was used for this purpose.

3.3 Collection of meteorological data

Meteorological data were collected at two meteorological stations; Pertanian Tanjong Karang (03˚25'08"N, 101˚11'17"E) and Mardi Tanjong Karang (03˚27'17"N, 101˚09'24"E); both situated between 8 to 12 km away from JSL (MetMalaysia, 2013). Two stations were used to compensate the different parameters available in each station.

3.3.1 Types of meteorological parameters used

The meteorological parameters used in this study are atmospheric pressure (hPa), rainfall (mm), atmospheric temperature (˚C) and relative humidity (%).

3.3.2 Instruments used for measuring meteorological data

A set of conventional and digital instruments were used to collect the meteorological parameters required for this study (**Plate 3.6**).

Atmospheric pressure - the aneroid barometer 6079 by Belfort Instrument was used to measure the atmospheric pressure. The clock faced instrument measures the pressure of air by the weight of column of air above it.

Rainfall –digital tipping bucket rain gauge model 52502 by Young Inc. was used to quantify the amount of rainfall at the station. The rain gauge readings were downloaded from the data logger on a weekly basis and analyzed for accuracy.

Atmospheric temperature – the dry bulb thermometer model 112 by Zeal, UK was used to determine the air temperature and not affected by the moisture in the air. In addition, the maximum and minimum thermometers model 6042 by Belfort Instrument were used to measure the maximum and minimum temperature within a day. All measurements were taken daily.

Relative humidity – the wet bulb thermometer is a moistened thermometer wrapped in a wet muslin cloth. The temperature readings from this thermometer and the dew point temperature from psychometric charts were used to compute the relative humidity of air.

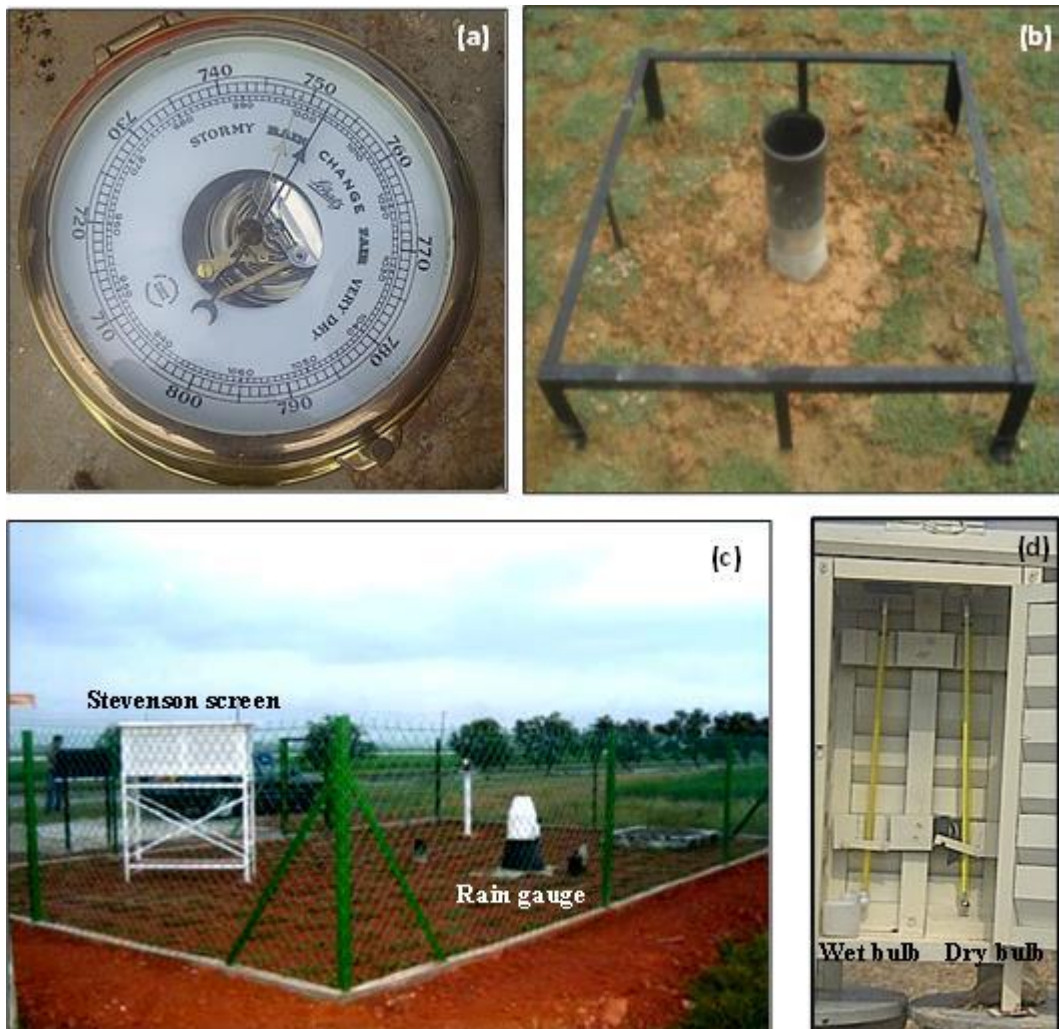All the thermometers used in this study were shielded from radiation and moisture inside a Stevenson Screen.

Plate 3.6 : Meteorological instruments (a) aneroid barometer; (b) check gauge; (c) Stevenson screen & rain gauge; (d) wet and dry bulb thermometer (MetMalaysia, 2013)

3.3.3 Duration of observation

The field measurements for this study were carried out for a period of six months on a weekly basis. The first phase was from October 2012 to December 2012 while the second phase was from May 2013 to July 2013. These two phases coincided with the rainy months and dry months at JSL.

3.4 Materials and methods used for Biocover

In this study, two materials that were used as Biocover are compost mixed with fresh brewery spent grain (BSG) and BSG compost.

3.4.1 Compost

Two different types of compost were used; (1) compost made from grass clippings and cow manure; (2) compost made from fresh brewery spent grain (BSG) and cow manure. The grass clippings were collected from the fields around the university compound. The freshly cut grass between 5cm to 7cm length were used immediately on the day it was cut to avoid any fermentation. While the cow manure was collected from nearby cow shed. The cow manure used was approximately one week old.

3.4.2 Fresh Brewery Spent Grain (BSG)

BSG is a by-product of the brewers and it represents about 85% of the by-products of a brewery (Mussatto *et al*., 2006). It is an organic waste from barley and other cereal malts used to produce beer (Thomas *et al*., 2006). BSG is available throughout the year in Malaysia and its main application is as animal feed due to the high protein and fibre content. However, it is under-utilized as only approximately 15% are sold for feedstock while the balance is discarded to landfills. For this study fresh BSG (**Plate 3.7**) was provided by Carlsberg Brewery Malaysia Berhad for the research purposes with no charges.

Plate 3.7: The texture of fresh BSG

3.4.3   Composting

3.4.3.1 Compost from grass clippings and cow manure

A compost mixture of 75% grass clippings and 25% cow manure of weight percentage were prepared in accordance to Navarani (2009), Sitiaishah (2011) and Jayanthi (2013) using ratio 3:1 grass clipping to cow dung. A heap of uniformly mixed grass clippings and cow manure as shown in **Plate 3.8** with height 1m and width 2m was prepared. The heap was mixed well to ensure even distribution of microbes for optimum composting. Water was added to the compost pile to maintain a moisture level between 50 to 60% throughout the composting process. Daily manual turning of the compost pile was done for the first 8 days to maintain an aerobic condition of the pile. Thereafter, it was only mixed every alternate day. Throughout the composting period the daily temperature

reading and moisture content of the compost pile were calculated. Temperature was taken using an electronic thermometer (Model Oregon Scientific SA880SSX) and moisture content was determined gravimetrically by oven drying compost at 104°C for 24 hours and expressed as the mass ratio of water to drying compost, following the ASTM (2004) procedure. The pH of the compost was measured using the Metter Toledo pH meter.

Plate 3.8: Compost pile from grass clipping and cow manure (before and after)

3.4.3.2 Compost from fresh BSG and cow manure

The ratio of BSG to cow manure was maintained at 3:1 to establish a standard comparison between both the compost. Similar method of preparation was done according to the grass clippings and cow manure compost. Temperature, pH and moisture content were measured accordingly. However at the beginning of the experiment the fresh BSG was spread out to cool down and to remove excess moisture. In both compost piles, it took 2 to 3 days to increase beyond mesophilic (10˚C to 40˚C) temperatures and reach the thermophilic (41˚C to 70˚C) stage of composting. Later it

decreased to ambient level and plateaued once it matured. Further chemical analysis of the matured compost was carried out in duplicates according to the standard procedures as described in the results to examine the physico-chemical properties of the compost.

Plate 3.9: Matured BSG compost

3.4.4 Batch experiment on CH_4 oxidation with different ratio of fresh BSG and compost

Batch experiments were carried out using Wheaton bottles. Mixture of fresh BSG and compost ranging from 10 to 100% ratio of BSG to compost with a 10% increase was prepared in 150ml bottles in triplicates. Each bottle with 20g of substrate (compost and BSG) was sealed with a septa and aluminium seal to ensure it is air tight (**Plate 3.10**). Then 18ml of air was withdrawn from the headspace of each Wheaton bottle using an airtight syringe and replaced with 6ml of CH_4 (99.9% purity) and 12ml of O_2 (99.98% purity). This amount provided a mixing ratio of approximately 4% of CH_4 (v/v) and 8%

O_2 (v/v) required for substrate saturation and determination of maximum CH_4 oxidation rate (Perdikea *et al.*, 2008). O_2 in the headspace maintains an aerobic condition within the bottles during the experiments.

Plate 3.10: Preparation of sample and Wheaton bottle experiments

Concentrations of CH_4, O_2 and CO_2 in the headspace were measured daily using the Shimadzu 8A Gas Chromatography (GC) equipment for the different ratios (**Plate 3.11** and **Plate 3.12**). Later, the procedures were repeated for the best chosen ratio and tested with different parameters such as temperature, pH, and moisture content.

Plate 3.12: Injection of gas sample to GC

3.4.5 Response surface modelling for parameter setting

The design experiment version 7 software was used to design the experiment setup for optimal temperature, pH and moisture content of the biocover material. Response surface modelling was used to improve the accuracy of the results.

3.4.6 Batch experiment on CH_4 oxidation using best ratio of BSG and compost

3.4.6.1 Influence of temperature on CH_4 oxidation

Batch experiment was carried out as described in Section 3.4.4 to test the influence of temperature on CH_4 oxidation between 25°C to 55°C with 5°C increase. Triplicates samples were incubated in different temperature setting and analyzed until CH_4 concentration becomes zero or reaches a plateau. The original temperature of mixture 30°C was used as a controlled experiment.

3.4.6.2 Influence of pH on CH_4 oxidation

Batch experiment was carried out as described in Section 3.4.4 to test the influence of pH on CH_4 oxidation by modifying pH of mixture ranging from pH4 to pH8. Sulphuric acid was added to lower the pH level while nitric acid to increase the level. The original pH 6 of the mixture was used as a control set up. Each pH was done in triplicate and headspace gas was analysed using GC. The analysis was carried out until the CH_4 concentration becomes zero or reaches a plateau.

3.4.6.3 Influence of moisture content on CH_4 oxidation

Batch experiment was carried out as described in Section 3.4.4 to test the moisture content on CH_4 oxidation using different moisture content. The original moisture content of the mixture was about 66% and it was used as a control setup. Moisture content is lowered by oven drying the mixture while it is increased by adding deionised water to the mixture. Moisture content of 50% to 80% was tested using triplicate bottles.

3.4.7 Batch experiment on CH_4 oxidation using composted BSG

Batch experiment was carried out as described in Section 3.4.4 using BSG compost. The procedures were repeated for temperature, pH and moisture content.

3.4.7.1 Influence of temperature on CH_4 oxidation

Batch experiment was carried out as described in Section 3.4.4 using BSG compost. The incubation temperature ranges were chosen similar to that in Section 3.4.4.1.

3.4.7.2 Influence of pH on CH_4 oxidation

Batch experiment was carried out as described in Section 3.4.4 using BSG compost. The pH was adjusted according to method in Section 3.4.4.2.

3.4.7.3 Influence of moisture content on CH_4 oxidation

Batch experiment was carried out as described in Section 3.4.4 using BSG compost. The moisture contents were adjusted similar to method in Section 3.4.4.3.

3.4.8 Column Experiment

The column experiments represented an advanced step for evaluating CH_4 oxidation of biocover material of certain thickness or height. Columns of one metre height were fabricated using 10mm thick PVC with an internal diameter of 0.15m. Sampling ports at 0.1m interval were embedded in the columns to enable gas sampling at different heights (**Plate 3.13**). The columns were prepared in duplicates and the experiment was repeated twice.

Plate 3.13: The PVC columns used for both laboratory and landfill experiment

3.4.8.1 Column experiment using fresh BSG and compost in laboratory condition

The best BSG to compost ratio with the optimum parameters from the batch incubation experiments were chosen for the column experiment. The biocover material (10 – 12kg) was placed inside the columns and the top of the column was sealed with a 5mm thick plexiglass to ensure air-tight. Similar to the batch incubation experiment approximately 4% of CH_4 (v/v) and 8% O_2 (v/v) was introduced from the bottom sampling point into the column using a Dwyer Rate-Master flow meter. The percentage of CH_4 and O_2 introduced were to obtain maximum saturation of the gases to induce CH_4 oxidation (Navarani, 2009; SitiAishah, 2011; Jayanthi, 2013). Later, the gas samples at each port were analysed using GC and results of CH_4 oxidation rate were recorded. This experiment was repeated with composted BSG. **Figure 3.3** shows the column experiment set up for laboratory condition.
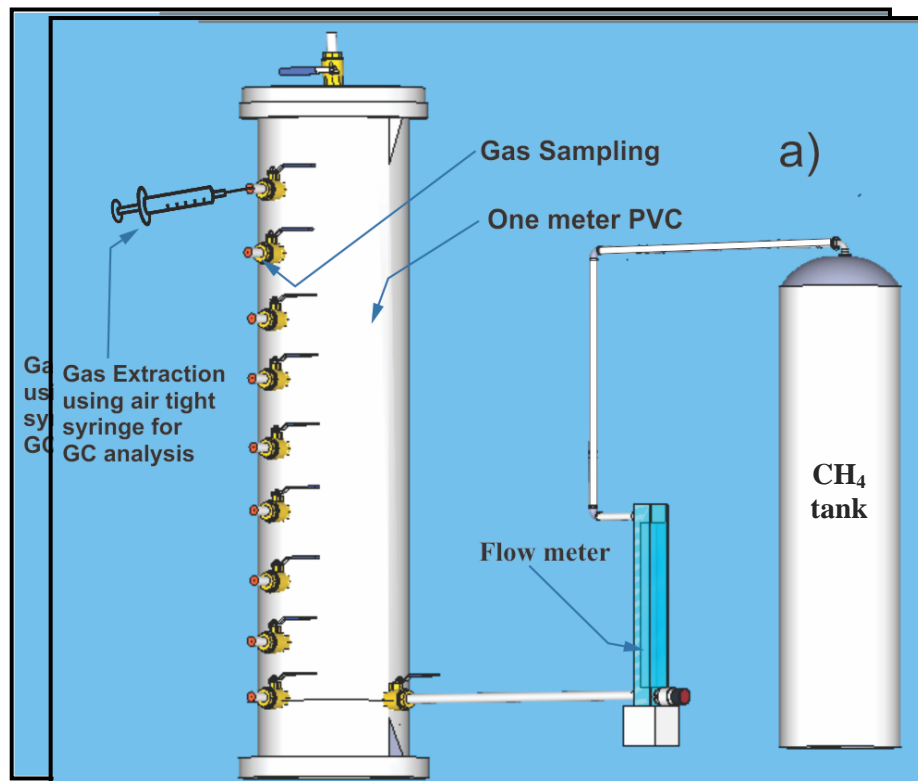

Figure 3.3: Column experiment setup in the laboratory

3.4.8.2 Column experiment using fresh BSG and compost in landfill condition

The column experiment was carried out in the landfill as described in Section 3.4.8.1. However, for CH_4 and O_2 supply, LFG and ambient air were used, respectively (as shown in **Figure 3.4**). The percentage of CH_4 form LFG was determined beforehand and the flow rate of the LFG is adjusted accordingly (**Plate 3.14** and **3.15**). O_2 input was also done in the similar method. Once the gases are supplied, the initial readings of gas samples were taken from each port using an air tight syringe and 10ml vacuum vial (**Plate 3.16** and **3.17**). Later, the gas samples were analyzed in the laboratory using GC. The GC was used to analyze the gas samples in both laboratory and landfill condition to ensure a uniform analysis method without any biasness in terms of instrument.
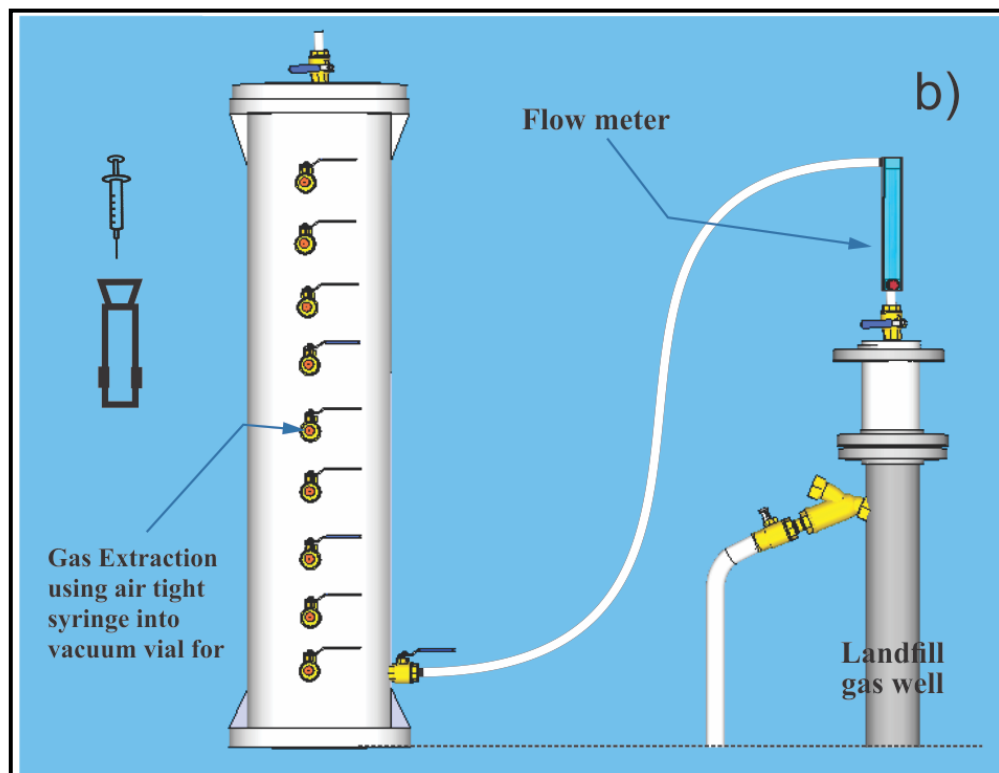

Figure 3.4: Column experiment setup in the landfill

Plate 3.15: The Dwyer Rate Master flow meter is adjusted according to the amount of CH_4 and O_2 supply needed
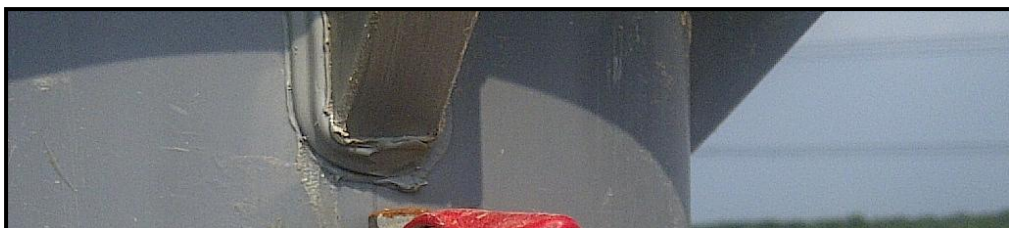
Plate 3.17: Gas sampling using air tight syringe and vacuum vials