# **CHAPTER III**

# **METHODOLOGY**



# 3.1 Runoff Samples

Runoff samples were synthesized. All metal solutions (Cd, Cu, Pb, and Zn) were prepared by dissolving metal nitrate salt in 1% nitric acid and fixing with concentrated nitric acid to make 100 ppm (part per million) stock solutions. The stock solutions were diluted with de-ionized (DI) water to obtain desirable metal concentrations. The pH of all samples was adjusted to 5 by diluted sodium hydroxide and nitric acid solutions.

# **3.2 Composted Material**

Grass clippings were collected form the soccer field of the Chulalongkorn University, Bangkok, Thailand. They were Manila grass (*Zoysia matrella Merrill*) (Figure 3.1). Their leaves are thin as shown in Figure 3.2. After collection, the clippings, whose size was 2-5 cm, were washed twice with tap water and once with DI water, consecutively. Then, they were air dried and used within 24 hours.



Figure 3.1 Manila grass.



Figure 3.2 Characteristic of manila grass leaves.

## **3.3 Experimental Procedure**

#### 3.3.1 Composting Process

Grass clippings were composted in bin composters, whose design was modified from a composting bioreactor developed by the Cornell Waste Management Institute (2003) as shown in Figure 2.1. A diagram illustrating the bin composter and its dimensions are shown in Figure 3.3. The actual bin composter is shown in Figure 3.4. Figure 3.5 shows the bottom of the bin composter.

The clean grass clippings were composted at 40, 50, 60, 70 and 80% moisture content and at initial pH 5, 6, 7, 8 and 9. Mass of grass in each composter was 300 g (wet weight). The composters were operated at room temperature and the content was mixed daily by stirring it with a rod. The composting period was 63 days following Paredes et al. (1996) and Tiquia et al. (2002). Moisture content was controlled according to the method described in Suehara et al. (1999) as described in the Analyses Section while the initial pH was adjusted using diluted HCl and lime solutions.



Figure 3.3 Bin composter diagram.



Figure 3.4 Bin composter.



Figure 3.5 The bottom of bin composter.

## 3.3.2 The Adsorption Isotherm Test

All isotherm tests were conducted at room temperature ( $25\pm2$  degree C).

## 3.3.2.1 Equilibrium time determination

Batch adsorption tests were conducted to examine the equilibrium time for heavy metal adsorption by grass clippings compost and noncomposted grass clippings (control). The noncomposted grass clippings were prepared according to the procedure described in Section 3.2. The grass clipping compost was washed with distilled water, dried at 60°C in a hot air oven, ground with a grinder and sieved pass 1 mm mesh before use. Approximately 85-95% by mass of the ground compost passed the sieve. Sorption equilibrium time was determined by the addition of 0.02 g of the compost (or grass clippings) to 25 mL of the metal solution in a 50 mL centrifuge tube at pH 5.0 (adjusted by diluted HNO<sub>3</sub> and NaOH solutions). The centrifuge tube was shaken at 100 rpm and a sample was removed at 3, 5 and 15 hours and filtered (Whatman 40). The filtrate was measured for the residual metal concentration. The equilibrium time was the time that there was no longer change in the residual metal concentration based on the concentration comparison with the previous sampling time.

## 3.3.2.2 Adsorptive capacity determination

At first, the mass of the compost was varied in the adsorption isotherm tests. The variation changed the sample pH, which could affect the adsorption. As a result, the tests were conducted following a 9-point isotherm technique by varying the concentration of the 25 mL metal solution. On the contrary, the control adsorption tests fixed the metal concentrations and varied the mass of non-composted grass. The 50 mL centrifuge tubes containing the sorbent and metal solution were shaken at 100 rpm. After the equilibrium time was reached, the sorbent was removed by filtration and the filtrate was analyzed for the metals. The adsorption capacity of the sorbent was determined based on the fitting of the data with the Freundlich model. Some compost and control samples were measured for CEC.

## 3.4 Analyses

Metal concentrations were analyzed using a Vista-MPXTM Inductively Coupled Plasma Optical Emission Spectrometer according to Standard Methods (1998). To determine the initial pH of the grass clippings, they were mixed with DI water at 1:10 w/v (clippings:water) and the pH of the mixture was measured using a pH meter (Sension<sup>TM</sup> model 51935-00). Moisture content measurement was according to the procedure described in Suehara et al. (1999) as follows. The weight of an aluminium foil cup (W<sub>0</sub>) was measured. A sample of the compost was transferred into the aluminium foil cup and the weight (W<sub>1</sub>) was measured. The compost in the cup was dried for 2 hours at 130°C, and the dried compost was cooled in a desiccator for 30 min, after which the weight (W<sub>2</sub>) was measured. The approximate moisture content (M<sub>ap</sub>) was calculated based on the following equations.

$$M_{ap} = 100(W_1 - W_2)/(W_1 - W_0)$$

To control the moisture content, additional water weight  $(W_{wdry})$ , that needs to be supplemented, was determined based on the values of  $M_{ap}$ , the total wet weight of composted grass  $(W_{wet})$  and the moisture content desired  $(M_{set})$  according to the following equation.

$$W_{wdry} = W_{wet} \left( M_{set} - M_{ap} \right) / (100 - M_{set})$$

The moisture adjustment was performed every 24 hours. Compost volume reduction was determined based on the compost height change in the composter and corresponding composter diameters (at initial and final heights). The CEC measurement was determined by the Ammonium Saturation and Distillation method (Method of Soil Analysis PartII.sssa.1965) at the Agricultural Academic Department, Kasetsart University, Bangkok, Thailand.