

Developing a Standard Method for Measuring Free Air Space of Compost Materials

By

Qunfeng Wang

A Thesis

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Master of Science

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**DEVELOPING A STANDARD METHOD FOR MEASURING FREE AIR SPACE OF
COMPOST MATERIALS**

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**A Thesis/Practicum submitted to the Faculty of Graduate Studies of The University
of Manitoba in partial fulfillment of the requirements of the degree
of**

Master of Science

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ABSTRACT

The composting process and compost materials have been long and extensively researched in chemical and biological properties. However, numerical and physical models of the composting environment require a better understanding of the independent physical properties: bulk density (BD), particle density (PD), total air space (TAS) and free air space (FAS). For windrow composting, FAS is one of the most important parameters indicating oxygen transportation, and it helps composting engineers to design and maintain a composting pile with sufficient aeration. Therefore, accurate FAS measurement is very important for windrow composting.

Currently, widely accepted and in-use FAS measurement methods include the particle density (PD) method and the gas pycnometer (GP) method. Recently, Test Method for Examination of Composting and Compost (TMECC) method: 03.01 A & B (US Composting Council, 2001), maximum moisture content (MC^{max}) method (McCartney and Eftoda, 2002) and dry-flow method (Micromeritics company, 2000) were reported to be able to measure FAS.

The purpose of this research was to develop a standard method for FAS determination that suits all composting materials. All available FAS methods were tested using different feedstock (different sizes of woodchips, leaves, woodchips/biosolids or leaves/biosolids) and analyzed individually for their

suitability and precisions. Method comparison was conducted based on the same particle size range woodchips (3.18 – 9.53 mm) and MC^{max} method was selected as the target FAS measurement method because: 1) its measured FAS is mostly close to the true value and 2) it has high precision and low cost.

The procedure of MC^{max} method was developed and standardized according to the standard method 1040: method development and evaluation (APHA 1995). The developed method was applied to different bulking agents (different sizes of woodchips, straw, leaves and wood shavings) and obtained precise FAS results (relative standard deviations ranged from 0.11 to 2.02 %). Unfortunately, it was found ineffective for determining FAS of composting mixtures due to the total solids loss during the operation was as high as 31.5 %. In order to overcome the shortage, a FAS numerical model that combines the MC^{max} method and Haug's conceptual model (Haug, 1993) was developed. In this model, FAS of the mixture was calculated from the FAS of bulking agents and other related parameters. For two kinds of woodchips/biosolids mixtures, FAS determined by the new model was approximately 12 % lower than that by PD method. For straw/biosolids mixture, the difference was high as 39 %. Reasons for these differences were the micro-pores ratio and other related physical properties of samples. The new FAS determination model showed its ability to determine FAS of composting mixture but further work is required to determine its accuracy and suitability for a broad range of materials.

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NOMENCLATURE AND ABBREVIATIONS

$^{\circ}\text{C}$	Degrees centigrade
A	Constant indicating the compressibility of the material
ANOVA	Analysis of variance
APHA	American Public Health Association
B	Constant indicating the magnitude of the bulk density
BD	Bulk density (wet basis)
BD_b	Bulk density of bulking agents
BD_{dry}	Dry bulk density
BD_{eff}	Effective bulk density
BS	Biosolids
d	Depth
ε	Total porosity
FAS	Free air space
FAS_b	FAS of bulking agents (fractional)
FAS_m	FAS of mixture (fractional)
Full-scale woodchips	Woodchips used for on-site research purpose
g	Gram
GP	Gas pycnometer
L	Liter
LV	Leaves
Lab-scale woodchips	Woodchips used for lab research purpose

m	Mass
m_b	Mass of bulking agents
m_{bs}	Mass of biosolids
m_m	Mass of mixture
m_t	Total mass
m_w	Mass of water
m_w^{\max}	Maximum mass of water
mL	Milliliter
MC	Moisture content
MC_b	Moisture content of bulking agents (fractional)
MC_{bm}	Moisture content of bulking agents in mixture (fractional)
MC_{bs}	Moisture content of biosolids (fractional)
MC_m	Moisture content of mixture (fractional)
MC^{\max}	Maximum moisture content (fractional)
MC_{sm}	Moisture content of substrate in mixture (fractional)
ODW	Oven dry weight
P	Pressure
PD	Particle density
PS	Pore space
θ	Volumetric content
ρ	Density

ρ_w	Density of water
R	Volumetric ratio
R_{bs}	Volumetric ratio of bulking agents to substrate
R_{mb}	Volumetric ratio of mixture to bulking agents
RSD	Relative standard deviation
S	Solids content
S_b	Solid content of bulking agent (fractional)
S_{bm}	Solid content of bulking agents in mixture (fractional)
S_{bm}^m	Minimum solid content of bulking agents in mixture (fractional)
S_m	Solid content of mixture (fractional)
STDEV	Standard deviation
TAS	Total air space
TS	Total solids
TMECC	Test method for the examination of composting and compost
USCC	US Composting Council
US-EPA	United States Environmental Protection Agency
V	Volume
V_a	Volume of air
V_B	Bulking volume of sample
V_c	Volume of cell
V_E	Expansion volume

V_s	Skeletal volume
V_t	Total volume
W	Weight
W_c	Weight of cell
W_p	Weight of empty pycnometer
W_s	Weight of sample
W_w	Weight of pycnometer filled with water
W_{sw}	Weight of pycnometer filled with saturated sample and water
WC	Woodchips

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*To all family members,
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Chapter 1

Introduction

As a popular way of waste management, the composting process has been studied extensively on its chemical properties: pH, nutrients, carbon to nitrogen ratio, maturity, etc. (Golucke, 1977; Zucconi, 1981; Haug, 1993; Ozores-Hampton, 2002; Carr, 2002) and microbiological properties: microorganisms and pathogens (US-EPA, 1980; Hay, 1985; Strom, 1985; Haug, 1993; Tiquia, 2002). However, a complete understanding of the composting process requires an understanding of the physical properties of the compost material involved. Furthermore, the increasing economic importance of compost materials together with their production, handling, storage, processing, quality evaluation, and marketing also demand a better knowledge of significant physical properties of these compost materials. In addition, microbial activities and oxygen transport of compost material are highly depended on its physical properties (Nakasaki et al. 1987; Rynk, 1992; Haug, 1993).

Generally, physical properties of compost material include shape, size, colour, appearance, density, and porosity. Among them, the most important physical properties affecting compost engineering may be bulk density (BD) and porosity.

BD of compost material is the ratio of mass to bulk volume. To complicate matters, BD varies with depth in a composting pile. Porosity usually includes

spaces occupied by air and water. Free air space (FAS), the air space between particles, is one of the most important parameters to determine oxygen transport.

Many factors affect BD and FAS of compost materials: MC, size, bulking agents, and even the depth in a compost pile. Among them, bulking agents play an important role in composting process by affecting BD and FAS, especially FAS. It has been verified that different types of bulking agents have different abilities to provide FAS (McCartney & Chen, 2000). FAS contribution by bulking agents includes two ways: 1) providing porous structure and 2) adsorbing water into micro-pores. Porous structure mainly depends on size and strength of particles, while water adsorption is controlled by internal structure of particles.

Measurement of BD and FAS has engineering and economical significances. Accurate measurement of BD is important to determine material volume and handling cost, while accurate measurement of FAS is critical to estimate the air movement through a composting pile.

Current available measuring methods for BD can be classified as two categories: 1) volume-weight method and 2) compressive cell method (McCartney and Chen, 2000). Volume-weight method has long been used (Haug, 1993; Chen, 1998; Mbah and Odili, 1998; USCC, 2001) but its measurement only represents the BD of surface sample. For this reason, the compressive cell method was developed to

measure BD of sample at any location in a pile by simulating the pressure conditions.

Although measuring methods of BD have become mature and clear, the concept of FAS and its effective measuring method are still ambiguous. Traditionally, the concept of FAS (inter-particle air space) and its determination method (particle density method) borrowed from the soil science field are widely adopted in composting engineering area (Leonard and Ramer, 1993; Das and Keener, 1997; Oppenheimer, 1997; Chen, 1998; Agnew and Leonard, 2002). However, PD method was found ineffective to measure FAS of compost materials because most of them contain porous bulking agents that have high ratio of micro-pore space causing the deviation of FAS measurement (Eftoda and McCartney, 2002). In practice, the FAS of compost materials measured by particle density method actually equals to total air space (TAS), which includes inter-particle voids and intra-particle voids. Considered the micro-structure, intra-particle voids can be further classified as open pores and closed pores (Webb, 2001). However, in a compost pile, air movement is controlled by inter-particle air voids but not intra-particle voids (Haug, 1993). Therefore, FAS is defined as inter-particle air voids in this paper.

As mentioned above, the PD method is a traditional FAS measurement method that has been widely accepted and used by the majority of researchers in composting engineering. Another popular method for FAS measurement is gas

pycnometer method due to its precision and quickness (Oppenheimer, 1997; Baker *et. al.*, 1998; Agnew & Leonard, 2002). Besides these two popular methods, a rapid method (TMECC method 03.01 A & B; USCC, 2001), maximum moisture content method (Eftoda and McCartney, 2002), and dry-flow method (Micromeritics Company, 2000) have also been reported as suitable FAS measuring methods. Excluding the dry-flow method, all FAS methods will be compared in this experiment.

A thorough literature review of BD and FAS measuring methods is presented in Chapter 2. Experiment materials and methods are presented in Chapter 3. The experimental results and discussion are presented in Chapter 4. Conclusions and recommendations are presented in Chapter 5.

Chapter 2

Literature review

This chapter first introduces the general background of the physical properties of compost materials and then focuses on factors affecting BD/FAS and their measuring methods. The objective of this chapter is to summarize the literature related to BD/FAS variations and their effective BD/FAS measuring.

2.1 Physical Properties of Compost materials

Physical properties are very important for compost materials because of their influences on the composting process and utilization of final products. Focused on the influence on composting process, some of the most important physical properties include particle size, porosity, moisture content, free air space, bulk density, and particle density. The literature review about how these properties influence the composting process is presented in Chapters 2.1.1 to 2.1.4.

Compost utilization is well known to improve soil quality as a supplier of plant nutrients. However, in many cases the physical properties of compost play an important role in soil conditioning (Dick and McCoy, 1993). Reported by US-EPA (1993), the amendment of the soil with compost changes the soil physical properties, including improvements of porosity, water retention, and soil

infiltration. Leonard and Ramer (1993) pointed out that the amendment of soil with compost results in decreased bulk density and improved porosity and water retention.

2.1.1 Particle Size

Particle size of compost material is one of the controlling factors to the structure of composting bulk. Size of compost material determines their porosity, structure, and texture, which have significant influences on aeration during composting (Rynk, 1992). Many experiments have shown that the use of large size bulking agent increases porosity and aeration and decreases the need for windrow turning (Haug, 1993; Reinhart and Trainor, 1995).

Particle size also affects microbial activities of compost material. Reported by Nakasaki et al. (1987), the availability of the substrate to microorganisms is a function of particle surface area, which is determined by particle size. Haug (1993) also pointed out that anaerobic condition would happen if the particle size of compost material is too small.

In addition, particle size reduction in a composting process has been verified to increase the water retention and total porosity of materials (Mbah and Odili, 1998).

2.1.2 Moisture Content

As a vital physical factor, moisture content is used for recipe design of composting feedstock and performance maintenance in a composting process. Rynk (1992) recommended that the preferred range of moisture content is 50-60 %. Golueke (1977) suggested a range of 55-60% be a general rule for most compost materials. If the moisture is too high, compost materials become easy to be compacted so that FAS were reduced in the pile. Also, excess water displaces air in FAS and thus anaerobic conditions may happen in some regions of the composting pile. When moisture content is above 60%, free air space in the compost matrix becomes limiting (Schulze 1961). Das and Keener (1997) investigated the moisture effect on compaction and air permeability of compost and found that the compact ability increases but FAS decrease with increasing moisture content. In addition, Senn (1971) verified that high moisture content could impede the composting process by inhibiting the temperature increase.

2.1.3 Porosity, Total Air Space (TAS), and Free Air Space (FAS)

The composting matrix is a network of solid particles that contain voids and interstices of varying size. Voids between and inside particles are filled with air, water, or a mixture of air and water. Porosity is defined as the ratio of void volume to total volume, while TAS is defined as the ratio of gas volume to total volume. Haug (1993) suggested that the air space in the cellular matrix of bulking

agents, i.e. intra-particle voids inside bulking agent particles, are of little value to the substrate and oxygen transportation in the composting matrix. Therefore, FAS was defined as the inter-particle air space.

In a compost matrix, porosity usually includes the air-filled space (TAS) and water-filled space. TAS consists of FAS and micro-pores. FAS refers to the inter-particle air space and micro-pores is the air space inside particles. The relationship among TAS, FAS, micro-pores, water, and solid content is illustrated in Figure 2-1.

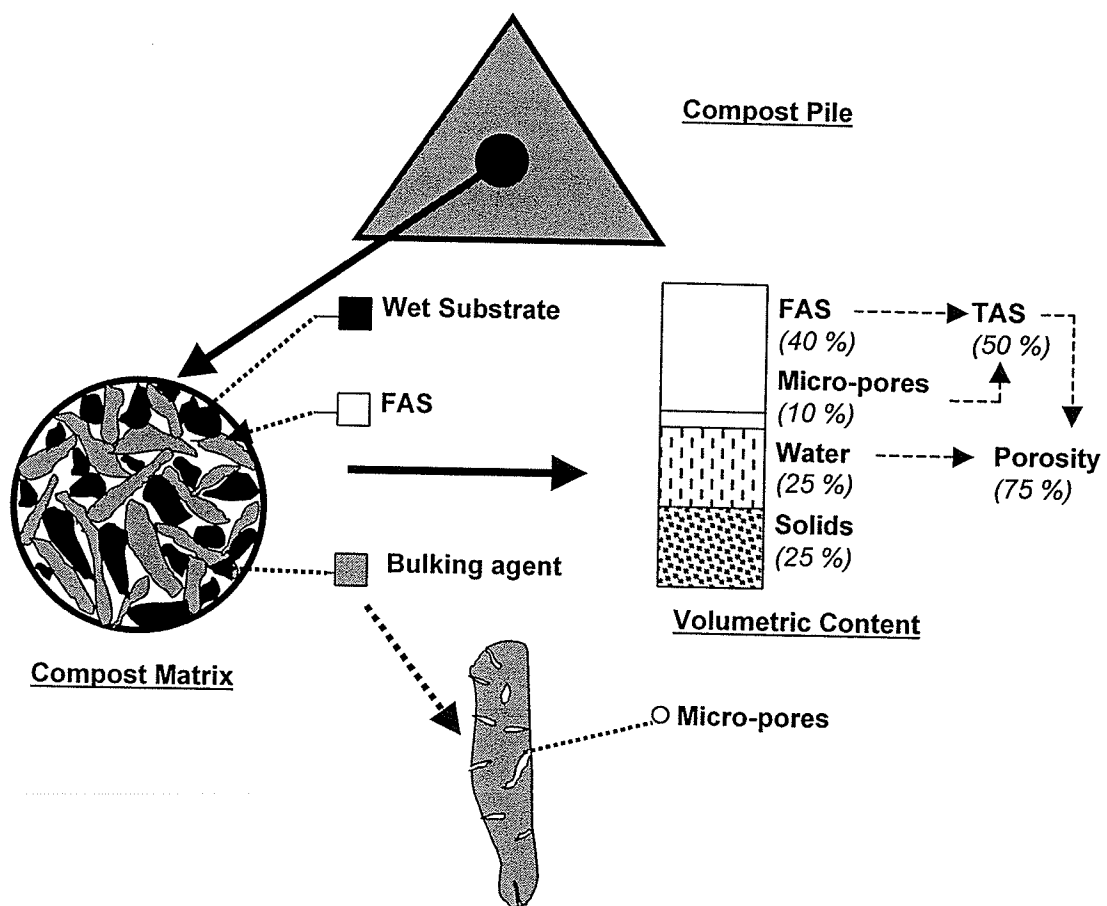


Figure 2-1 Schematic illustration of a compost matrix

According to the relationship illustrated in Figure 2-1, the FAS decreased with the MC, and it was confirmed by the following formula developed by Leonard and Ramer (1993):

$$\text{FAS} = \text{Porosity} (1 - M) \quad [2-1]$$

Where FAS = free air space ($\text{cm}^3 \text{cm}^{-3}$); M = moisture content (% wet basis);

Porosity = the space inside and between particles ($\text{cm}^3 \text{cm}^{-3}$).

Haug (1993) cited and recalculated the analytical data from Schulze (1962) to obtain the porosity and FAS results of several compost materials, which are shown in Table 2-1. The results indicated that different compost materials had different porosity and FAS and the FAS of dry materials were higher than those of wet ones.

Table 2-1 Analytical data of porosity and FAS for raw materials

Items	Moisture Content (%)	Porosity (%)	FAS (%)
Ground Garbage	63	77.7	27.4
Moist sludge cake	72	87.1	39.6
Dry sludge cake	6.0	74.3	72.0
Dry compost	10	83.3	80.4
Shredded paper	8.0	97.7	97.5

Source: From Haug, 1993

Webb (2001) pointed out that micro-pores inside porous particles include open-pores and closed pores. Considering the micro-structure, the distribution of TAS in a porous material is classified as the following:

- Inter-particle voids (FAS)
- Open pores
- Closed pores

As mentioned above, inter-particle voids (FAS) control the air movement but not open or closed pores. Therefore, the relationship between TAS and FAS can be expressed as following:

$$\text{FAS} = \text{TAS} - \text{open pores} - \text{closed pores} \quad [2-2]$$

Where FAS = free air space ($\text{cm}^3 \text{cm}^{-3}$); TAS = total air space ($\text{cm}^3 \text{cm}^{-3}$); open pores = pores inside a particle with outside connection ($\text{cm}^3 \text{cm}^{-3}$); and closed pores = pores inside particle without outside connection ($\text{cm}^3 \text{cm}^{-3}$).

2.1.4 Particle Density and Bulk Density

Particle density refers to the density of the solid particles collectively, and it is expressed as the ratio of mass of solid particles to volume of solid particles excluding the pore spaces between and within the particles (Black 1965).

Interrelationships of porosity, bulk density, and particle density can be expressed as following:

$$\text{Porosity} = (1 - \text{dry BD} / \text{PD}) \quad [2-3]$$

Where Porosity = the fractional total pore space (%); dry BD = bulk density of completely dried sample (g cm^{-3}); and PD = density of solid particles (g cm^{-3}).

Bulk density is defined as the ratio of the mass of oven-dried solids to the bulk volume of solids plus pore space at specified soil water content (Carter 1993). US composting council (1997) defines bulk density of compost as weight per unit volume of compost, calculated and reported on an oven dry ($70 \pm 5^\circ\text{C}$) weight basis.

In practice, particle density and bulk density have widely used value. Bulk density is needed for converting water percentage by weight to content by volume and estimating the weight of a volume of soil too large to weigh conveniently, such as the weight of a furrow slice, or an acre-foot (Black 1965). Particle density can be used for calculating porosity and TAS when the bulk density is known.

2.2 Factors Affecting BD/FAS Variations

Bulk density and free air space of compost material may vary depending on the moisture content of compost feedstock, type and amount of bulking agent, and the location in a compost pile.

2.2.1 BD/FAS Variations by Moisture Content

During the composting process, BD/FAS changes with many physical properties and moisture content was proved to be the most important one.

Oppenheimer *et al.* (1997) used the gas pycnometer to measure the air-filled porosity of dog food/woodchips and sewage sludge/woodchips at different MC. The author concluded that the air-filled porosity decreased with the MC and a linear relationship existed between them (results showed in Figure 2-2). Using similar theory, Agnew and Leonard (2002) conducted the research of FAS and BD of manure compost and MSW compost, and the results indicated that the FAS decreased but BD increased with the MC. More important, the linear relationship between FAS and BD was found:

$$\text{FAS} = 100 - 0.09\text{BD} \quad [2-4]$$

Where FAS is a percentage and BD is the wet bulk density (kg m^{-3})

Day *et al.* (1998) conducted an investigation of chemical and physical changes occurred during commercial composting and found that bulk density decreased but air voids increased with composting time. The authors pointed out that these changes of bulk density and air voids was attributed to the change of moisture content due to the dehydration of the material at the composting facility.

Mbah and Odili (1998) studied the variations of porosity and air space of five waste materials during composting process, which indicated that the air space decreased with time just because the water retention increased after composting.

Carter (1993) pointed out that the rate of change of soil bulk density depends on its structure and water content. This also confirmed that the moisture content is a very important parameter affecting the BD and FAS.

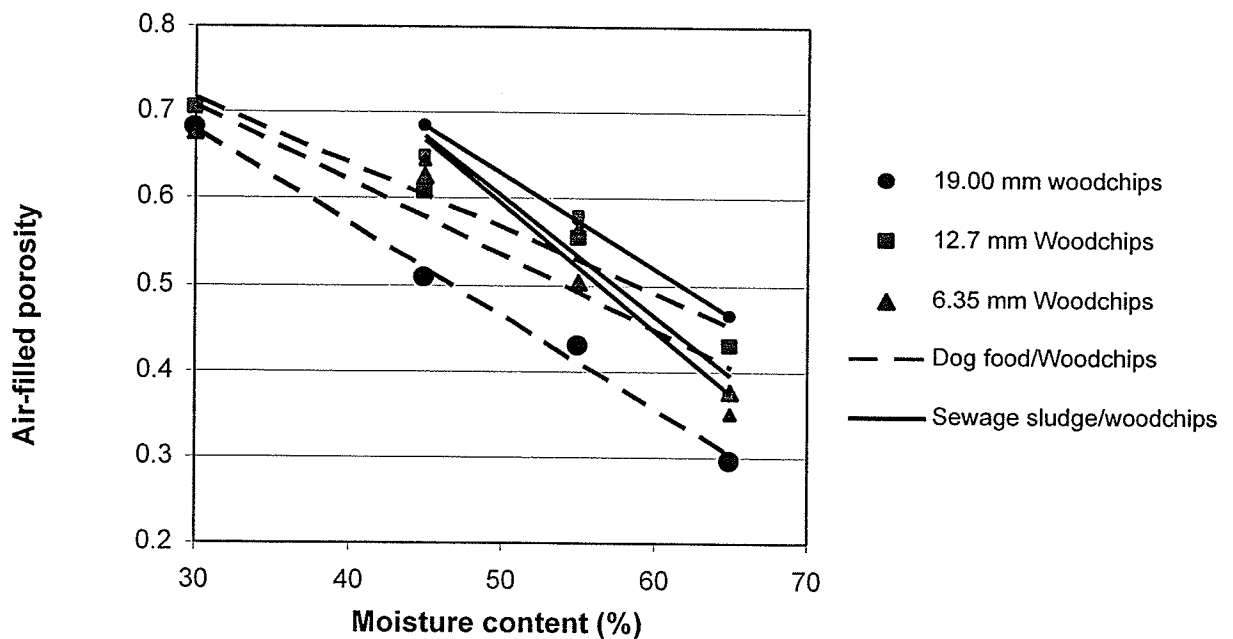


Figure 2-2 Air-filled porosity of dog food/woodchips and sewage sludge/woodchips at different MC and different size of woodchips (Oppenheimer *et al.* 1997)

2.2.2 BD/FAS Variations by Bulking Agents

Generally, wet substrates (e.g. sludge and wet manures) have high MC and their FAS can reasonably be assumed to be zero (Haug 1993). Therefore, bulking agents are required in a composting mixture to improve its FAS by providing structure support and moisture adjustment.

Numerous bulking agents can be used in practice, including woodchips, straw, wood shavings, rice hulls, shredded tires, and other materials. The selection of bulking agents is based on their capacity of structure support and water absorption. Large size and high strength of bulking agents are good for the structure support and high ratio of micro-pores inside bulking agents are important for water absorption capacity.

BD/FAS Affected by Types of Bulking Agents

Different types of bulking agents have different individual bulk densities and capacities on structure support and water-absorption, so BD and FAS of the mixture of substrate and bulking agents are very different.

It has been reported by McCartney and Chen (2000) that different bulking agents cause different BD and FAS of compost mixtures with the same moisture contents.

The investigation was conducted on woodchips-biosolids, straw-biosolids, leaves-biosolids, and ceramic-biosolids with same initial MC of 55 %, and results showed that dry BD were 0.28, 0.14, 0.22, and 0.66 g cm⁻³, respectively. As shown in Figure 2-3, different materials had different initial and compacted FAS. As the stress increased, compaction made FAS decrease and FAS of straw/biosolids and leaves/biosolids dropped more quickly than others did due to their bulk properties, such as strength.

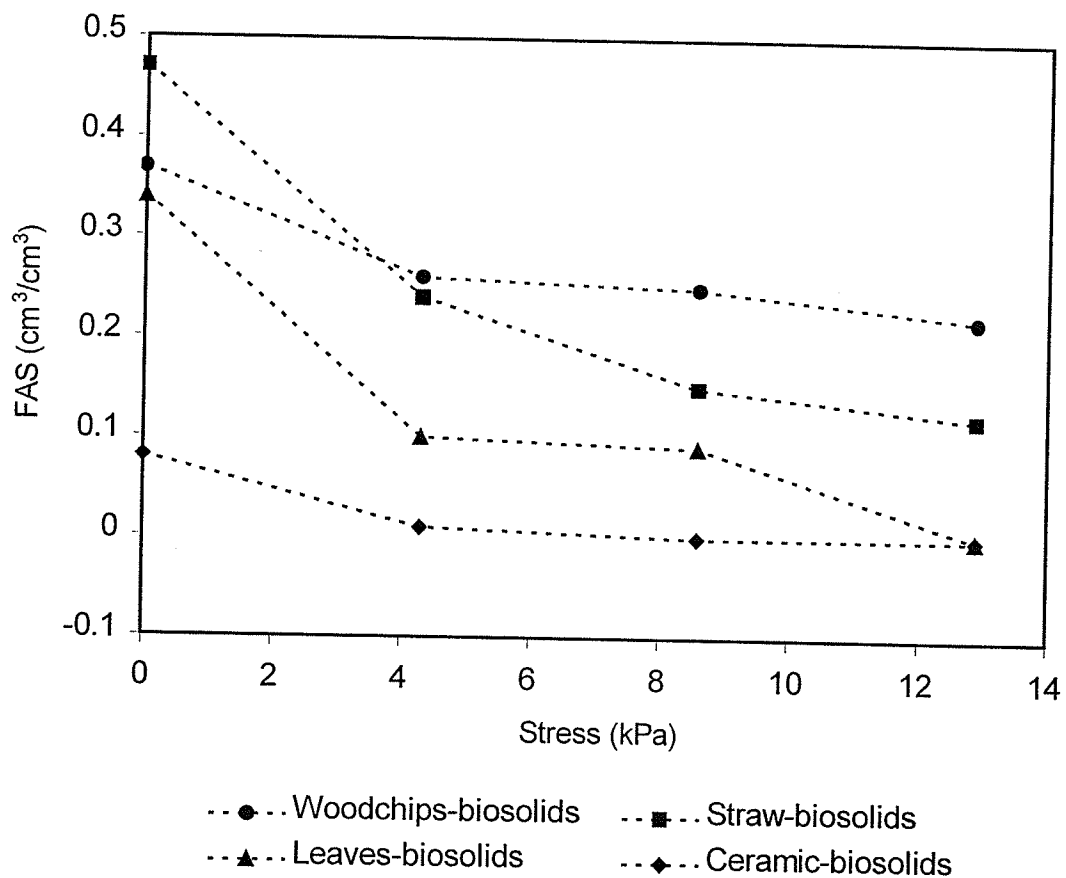


Figure 2-3 The effect of stress on the FAS (McCartney & Chen, 2000)

BD and FAS Affected by Ratio of Bulking Agents to Substrate

BD and FAS are also affected by the amount of bulking agents added in the compost mixture. Experimental results (Table 2-2) from Eftoda and McCartney (2002) indicated that BD and FAS were related to the ratio of woodchips to substrate in biosolids/woodchips mixtures. Specifically, BD of composting mixture decreased with increasing woodchips (WC) content, but FAS increased with increasing WC content.

Table 2-2 BD and FAS variations with ratio of woodchips to biosolids

Ratio of woodchips to biosolids	Wet Bulk density (g cm ⁻³)	FAS (%)	
		Unloaded	Loaded
1:1	0.654	27.4	0.0
2:1	0.496	40.0	7.5
3:1	0.419	45.9	31.2
4:1	0.392	47.1	33.6

Source: From Eftoda and McCartney, 2002

BD and FAS Affected by Particle Size and Species of Bulking Agents

For improving porosity and FAS of composting mixtures, large size bulking agents are better than small size ones because of the former can provide more porous structure. Wizibicki (2002) investigated the FAS of coarse (< 38.1 mm) and fine

(< 19.05 mm) woodchips and verified that FAS of coarse woodchips (62 %) was a little higher than that (60 %) of fine ones.

As one of the two functions of bulking agents, water absorption capacity is depended on internal micro-pores of particles. As the cellularities (or internal structure) of bulking agents processed from diverse raw materials are not unique, it goes without saying that the different types of bulking agents, e.g. woodchips, wood shavings, straw, etc., have different water absorption capacity. Even the same type of bulking agents (e.g. woodchips) processed from different species (e.g. poplar, pine, spruce, etc.) will also have different water absorption capacities.

During the water absorption process, maximum moisture content is reached when all spaces in the wall and lumina of woodchips are filled. Panshin & Zeeuw (1980) pointed out that the maximum MC usually ranges from 60 % (dry basis) for heavy woods (high specific gravity) to 200 % (dry basis) for the light woods (low specific gravity). This research revealed that the different wood species have different water-absorption and water retention capacities.

2.2.3 BD/FAS Variations in Compost Pile

In a real compost pile, the distribution of FAS and BD is not homogeneous because of compressive settlement caused by compost material itself. Therefore, the upper part of compost pile usually has more FAS and low BD than the lower part does. The force analysis of the compost material in a real pile (Figure 2-4) is helpful to explain BD/FAS variations with depth. For convenience, the compost located in the core cylinder area was adopted to perform the force analysis. The pile was assumed as 3.0 meter high and divided into 6 layers. As shown in Figure 2-4, 1st layer is on the top and does not receive any pressure, so the BD is lowest and FAS is the highest. However, the 4th layer received compressed force caused by the weights of 1st, 2nd, and 3rd layer so that the 4th layer will have higher BD and lower FAS than those of any other upper layers.

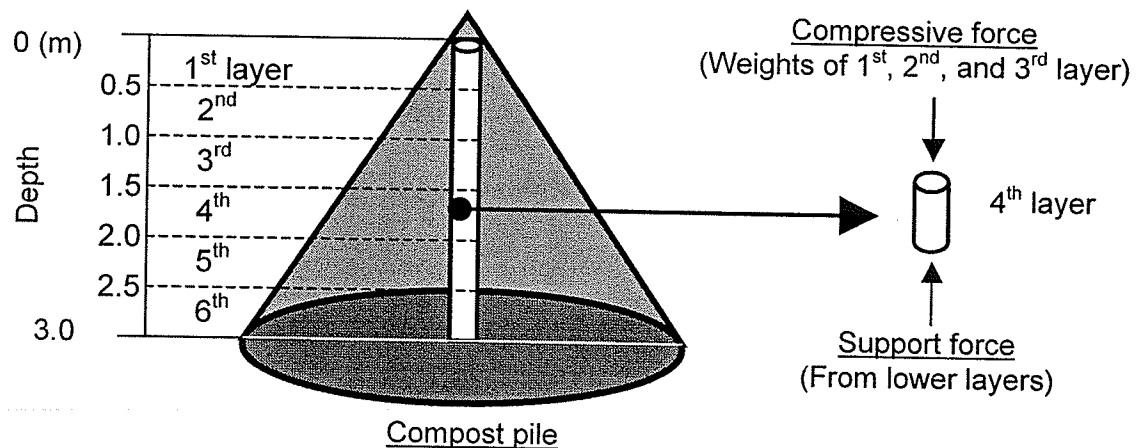


Figure 2-4 Force analysis of compost material in a real compost pile

Based on the above analysis, it is clear that the compressive force from the weight of the compost material causes variations of BD and FAS with depth in a compost pile. In order to quantify the BD and FAS change caused by the compressive force, compressive cell method (McCartney and Chen. 2000) and “effective particle density” method (Schaub-Szabo and Leonard, 1999) were developed to determine BD and FAS at any depth in a real compost pile by simulating the pressure condition. The detailed methodology of BD and FAS measurement in a compost pile will be discussed in section 2.3.

Schaub-Szabo and Leonard (1999) investigated wet BD of compost and peat in a pile and results showed that wet BD increased with depth. More recently, Agnew and Leonard (2002) applied the same methodology to determine wet BD of manure compost and MSW compost and obtained the same trend.

Using the compressive cell method to simulate pressure conditions in a real compost pile, McCartney & Chen (2000) investigated FAS of compacted compost materials and developed the following formula to calculate FAS of compacted materials:

$$FAS_i = \frac{FAS_0 \cdot V_0 - (V_0 - V_i)}{V_i} \quad [2-5]$$

Where FAS_i is the compacted FAS at depth i (m^3/m^3); FAS_0 is the original FAS at depth zero (m^3/m^3); V_0 is the volume of compost material at depth zero (m^3); and V_i is the volume of compacted material at depth i (m^3).

Results by McCartney and Chen (2000) (Figure 2-3) suggested that the FAS of compost materials decreased with increased stress. Since stress is in direct ratio to depth in a pile, FAS of compost materials actually decreased with the depth.

2.3 Methods for Measuring BD/FAS

2.3.1 Methods for Measuring BD

Methods for measuring bulk density generally include *in-situ* and *ex-situ* methods. In soil science field, *in-situ* bulk density measuring methods usually include clod method, core method, and excavation method, but few of them can be directly applied in compost materials. Based on the characteristics of compost, current bulk density measuring methods include two *ex-situ* approaches: 1) volume-weight method and 2) compressive cell method (McCartney and Chen 2000). The former was a traditional method used for many years and the latter was just developed within recent years (McCartney and Chen, 2000; Schaub-Szabo and Leonard, 1999; Das and Keener, 1997).

2.3.1.1 Volume-weight Method

In the volume-weight method, samples are collected in a certain container with known volume and BD is calculated by weight and volume of sample. Volume-weight method was widely used for BD measurement of soil, grain, and compost by many researchers because its easy-operation, quick, and low cost (Carter, 1993; Rameshbabu *et al.* 1996; Day *et al.* 1998; USCC, 2001).

However, there were few standard procedures of this method for compost materials. The only one found was the test methods for the examination of composting and compost (TMECC) developed by USCC (2001). Although TMECC method is designed as an official standard method, it was not widely applied and still in developing process. Other existing standard methods for BD were only found for peat (ASTM, 1994) and grain (Canadian Grain Commission, 1984).

Unfortunately, results from the volume-weight method only represent the BD of surface samples. Obviously, it is not suitable for accurately determining the BD of compacted compost in a pile because the reposition of sample in a measuring container will change its original structure and volume (Schaub-Szabo and Leonard, 1999). For this reason, compressive cell method (McCartney and Chen, 2000) and effective BD calculation model (Schaub-Szabo and Leonard, 1999) were developed, respectively.

2.3.1.2 Compressive Cell Method

Compressive cell was the container that initially developed by McCartney and Chen (2000) to investigate compost physical properties variations in a composting pile. A schematic description of the compressive cell is given in Figure 2-5. Using compressive cell method, BD of compacted compost in a pile can be determined and the relationship between dry BD and depth found to fit the simple power function:

$$BD_{dry} = Ad^B \quad [2-6]$$

Where, BD_{dry} is the dry bulk density (kg m^{-3}); A is the constant indicating the magnitude of the bulk density; B is the constant indicating the compressibility of the material; and d is the depth (m).

Using similar theory, Schaub-Szabo and Leonard (1999) proposed the concept and calculation model of effective BD. Effective BD was defined as the BD which, if constant with depth, would give the same area under a BD-depth curve as the measured values. It is expressed mathematically as the following:

$$BD_{eff} = \frac{1}{d} \int_0^d BD \, dz \quad [2-7]$$

Where, BD_{eff} = effective bulk density (kg m^{-3}); d = depth (m); BD = bulk density at any depth (kg m^{-3}). BD-depth curve was established on BD determined at any depth in a pile. The calculation model of BD is described as the following:

$$BD_n = [m_t - (m_c + \sum_{i=0}^{n-1} m_{mi})] / V_h \quad [2-8]$$

Where BD_n is the bulk density of compost material in layer #n (kg m^{-3}), m_t is the total mass of the material and containers (kg), m_c is the mass of the container (kg), m_{mi} is the mass of material in layer i (kg; supposed to increase when sample is refilled to original fill line), and V_h is the Volume to fill line (m^3).

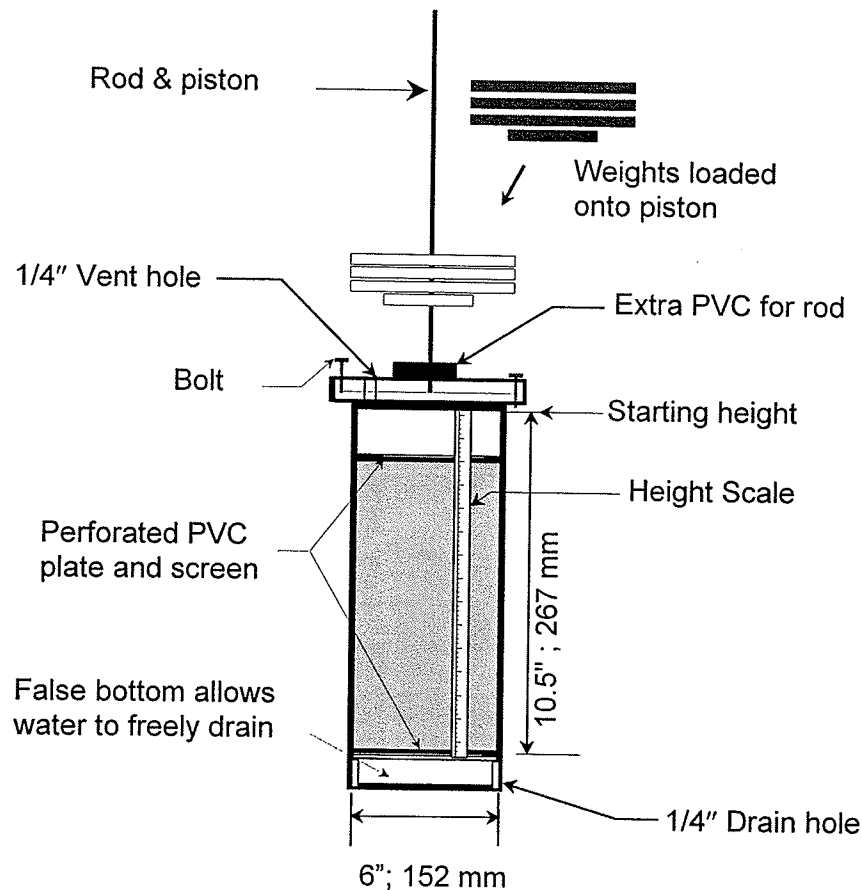


Figure 2-5 Compressive cell used for loaded and unloaded BD test
(McCartney and Chen 2000)

The development of compressive cell provided a physical model to estimated BD variations in a compost pile. The proposition of the concept and calculation model

of effective BD enhanced the understanding of BD variations in a compost pile provided a mathematical model to determine BD representing a whole pile.

2.3.2 Methods for Measuring FAS

Literature research found five available methods for measuring FAS, which include particle density (PD) method, gas pycnometer (GP method), TMECC method, maximum moisture content (MC^{max}) method, and dry-flow method. These methods were classified into two categories: 1) in-use methods and 2) other available methods. In-use methods refer to methods that have been long applied by the majority of researchers. As widely accepted methods, traditional PD method (Rynk 1992; Leonard and Ramar, 1993; Haug, 1993; Das and Keener, 1997; McCartney and Chen, 2000) and popular GP method (Day, 1964; Oppenheimer et. al., 1997; Baker et. al., 1998; Agnew and Leonard, 2002) are classified in this category. Other available methods include recently developed new methods and some potential methods.

2.3.2.1 In-use Methods

Particle Density Method

In the traditional soil science FAS method, measurement of particle density is the most important and complex step to determine porosity and FAS. For this reason,

the traditional FAS determination method was defined as “particle density (PD) method”.

As mentioned earlier, PD method was widely applied by the majority of compost researchers. McCartney & Chen (2000) investigated the BD and FAS of several compost mixture and used the following total porosity and FAS calculation models:

$$\varepsilon = 1 - \frac{BD_{dry}}{PD} \quad [2-9]$$

$$\theta = \frac{MC \cdot BD}{\rho_w} \quad [2-10]$$

$$FAS = \varepsilon - \theta \quad [2-11]$$

where ε = total porosity ($\text{cm}^3 \text{ cm}^{-3}$); θ = volumetric water content ($\text{cm}^3 \text{ cm}^{-3}$); FAS = free air space ($\text{cm}^3 \text{ cm}^{-3}$); BD_{dry} = dry bulk density of the sample (g cm^{-3}); BD = wet bulk density of the sample (g cm^{-3}); PD = particle density of the sample (g cm^{-3}); ρ_w = density of water (g cm^{-3}); and MC = moisture content of the sample (wet basis; fractional).

Gas Pycnometer (GP) Method

Day (1964) pointed out that attempts to measure porosity using water to fill the void space have proved ineffective because of erratic and inconsistent results. Therefore, a gas pycnometer method was recommended for porosity measuring.

The theory of GP method is Boyle's gas law, and a conceptual GP model (Day, 1964) was described in Figure 2-6. The tank 1 is empty for holding the air coming in, while the tank 2 is for porous material. Initially, valve 1 is open while valve 2 and 3 are closed, and air comes into tank 1. When certain pressure has been reached, valve 1 is closed. Secondly, open the valve 2, so the air can be distributed in tank 1 & 2. The volume of air in porous material in tank 2 and the porosity of the tested sample can be calculated according to the gas law. By the gas law, $m_1 = P_1 V_1 / R_1 T_1$; $m_2 = P_2 V_2 / R_2 T_2$; $m_3 = P_3 V_3 / R_3 T_3$; $m_3 = m_1 + m_2$, $V_3 = V_1 + V_2$, and $R_1 T_1 = R_2 T_2 = R_3 T_3$. Therefore, air-filled porosity can be calculated as the following:

$$P_1 V_1 + P_2 V_2 = P_3 V_3 = P_3 (V_1 + V_2) \quad [2-12]$$

$$V_2 = [(P_3 - P_1) / (P_2 - P_1)] V_1 \quad [2-13]$$

$$\text{Air-filled Porosity} = V_2 / V_1 = (P_3 - P_1) / (P_2 - P_1) \quad [2-14]$$

Here, P_1 , V_1 , and m_1 are, respectively, the absolute pressure, the volume occupied by the air and the mass of the air in tank 1 with valves 1 and 2 closed. P_2 , V_2 , and m_2 are, respectively, the absolute pressure, the volume occupied by the air and the mass of the air in tank 2 with valves 2 and 3 closed. P_3 , V_3 , and m_3 are, respectively, the absolute pressure, the volume occupied by the air and the mass of the air in the system with valves 1 and 3 closed and valve 2 opened.

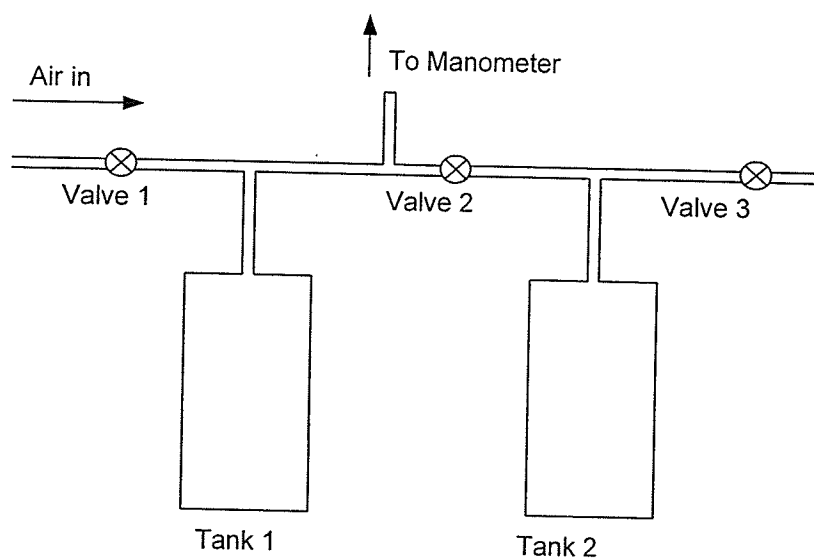


Figure 2-6 Apparatus used for determining voids in porous material (Day, 1964)

Currently, commercial gas pycnometers, as shown in Figure 2-7, are widely used for porosity determination of grain and soil. Although it provides a fast method to measure air space in porous material, its small chamber size and dry test material requirement limit its wide application on compost materials.

In order to overcome the shortage of commercial gas pycnometer, modified gas pycnometer with larger holding capacity was developed to measure the porosity of organic matrices (Oppenheimer *et al.* 1997) and compost materials (Agnew and Leonard, 2002). Results from the both experiments indicated that the gas pycnometer is reliable and precise for air-filled porosity measurement.

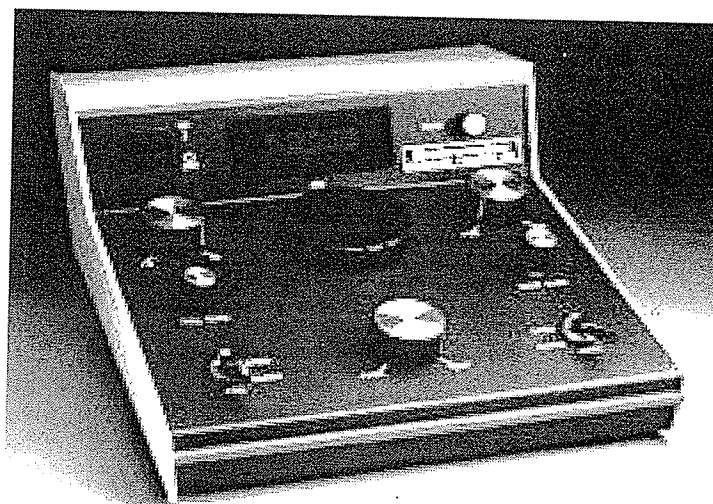


Figure 2-7 Commercial gas pycnometer for porosity measurement
(Micromeritics Company, 2000)

2.3.2.2 Other Available Methods

Beside the currently in-use FAS methods, other three methods were reported able to measure FAS: TMECC method 03.01-A and B (USCC, 2001), MC^{max} method (Eftoda and McCartney, 2002) and dry-flow method (Micromeritics Company, 2000).

TMECC Method 03.01-A and B

TMECC Method 03.01-A and B: quick-test for bulk density, porosity/pore space, free airspace and water-holding capacity of compost was developed by US Composting Council (2001). This method is developed to determine porosity and FAS of compost and its principal is air replacement by water. A sample of known

volume and mass is transferred to graduated beaker and saturated with water, and then excess water is drained. Changes in compost volume and mass, and the ratio of water retained relative to the amount of drained water provide a means for estimating compost porosity/FAS. FAS can be expressed as following:

$$FAS (\%) = (W_{before\ drain} - W_{after\ drain}) / V \quad [2-15]$$

where $W_{before\ drain}$ = weight of sample and water before drain (g); $W_{after\ drain}$ = weight of water-saturated sample after drain (g); and V = initial volume of sample (cm³).

Although TMECC method 03.01 A & B was a potential method for FAS determination, there is no application of the method reported in the literature.

Maximum Moisture Content (MC^{max}) Method

MC^{max} method is a new conceptual method proposed by Eftoda and McCartney (2002) for accurate FAS measurement when traditional PD method was found unable to determine FAS. It was found that FAS measured by PD method actually includes the micro-pores that are not considered useful to oxygen transportation. Also, authors defined FAS by PD method as total air space (TAS) in order to differ it from true FAS.

In MC^{max} method, maximum moisture content was reached after water-saturation of sample in boiled water. Air volume in micro-pores is determined by the volume of water hold inside sample particles. Therefore, FAS of sample can be calculated as the following:

$$\text{FAS} = \text{TAS} - \text{volume of micro-pores} \quad [2-16]$$

MC^{max} method was successfully used to determine FAS of woodchips (Eftoda and McCartney, 2002) and it has the potential to determine FAS of all compost materials. The detailed method was presented in section 3.1.4.4.

Dry-flow Method

Micromeritics' GeoPyc Model 1360 is the only known-of commercial instrument that automatically determines the envelope volume and density of a solid object by displacement of a solid medium. In this method, volume of inter-particle voids (FAS) can be calculated as:

$$\text{FAS} = \text{bulk volume} - \text{envelope volume} \quad [2-17]$$

Where, FAS = free air space or inter-particle voids (cm³ cm⁻³); bulk volume = the volume of particles, inter-, and intra-particle voids (cm³); and envelope volume = the volume of particles and intra-particle voids (cm³).

Although this instrument showed an easy way to measure FAS, there is no compost applications of the instrument reported in the literature. It offers future potential for FAS measuring.

2.3.2.3 Theoretical Comparison of All FAS Methods

Although all five methods were developed for measuring FAS, what is actually measured may be different among the methods (Eftoda and McCartney, 2002; Webb, 2001; TMECC, 2001). As mentioned previously, total air space (TAS) in a composting matrix includes inter-particle voids, open pores, and closed pores (Webb, 2001). Based on this classification, a theoretical comparison of air space included in the various FAS methods is presented in Table 2-3.

Table 2-3 Comparison of theoretical measurements of all FAS methods

Methods	Air space included (Y = Yes and N = No)		
	Inter-particle voids	Open Pores	Closed pores
PD	Y	Y	Y
GP	Y	Y	N
TMECC	Y	N	N
MC ^{max}	Y	N	N
Dry-flow	Y	N	N

From Table 2-3, air space measurements by both PD and GP methods should be greater than those measured by TMECC, MC^{max}, and dry-flow methods. Due to the absence of a standard method, a true FAS measurement of porous materials was unknown. By definition, in this thesis, FAS is a measurement of inter-particle voids only, therefore measurements by TMECC, MC^{max}, or dry-flow methods would be expected to most closely represent FAS.

To quantitatively compare different methods, a true value of FAS had to be assumed. In this experiment, an assumed true value was selected from the measurements by TMECC, MC^{max} , and dry-flow method according to their suitability, precision, and effectiveness.

2.4 Summary of Literature Review

The literature shows that BD and FAS are two of the most important physical properties of compost materials. Accurate measurements of BD and FAS have engineering significances. The former is helpful to estimate material volume and handling cost and the later is important to forecast the oxygen movement in a compost pile.

Both BD and FAS change during a composting process and are controlled by properties of composting feedstock and position in the pile. The majority of research pointed out that the most important factors affecting the BD and FAS are MC, particle size, and the water-adsorption capacity and the strength of bulking agents.

Currently, the BD measuring methods are becoming mature and complete as the development of compressive cell method and proposition of effective BD.

However, the concept and measuring method of FAS are still ambiguous. Although many methods were reported for FAS measurement, few of them are widely accepted and applied in practice. Even for the traditional PD method, measured FAS was found to deviate from the true value due to the high ratio of micro-pores of compost materials. For other available methods, their effectiveness was unknown and should be investigated.

Based on the above statement, it is necessary to compare current available methods for measuring FAS for their accuracies and suitability. An effective and efficient method can then be selected for accurate FAS measurement of all compost materials.

Chapter 3

Research Objectives and Experimental Techniques

The purpose of this research was to develop a standard FAS method that is suitable and effective for all compost materials. The research was conducted as three phases: 1) phase I: evaluating different methods and selecting an optimum one for standardization; 2) phase II: developing a standard MC^{max} method; and 3) Phase III: solving the limitations of the standardized MC^{max} method. The materials and methods used in each phase are described separately.

3.1 Objectives and Techniques of Phase I

As an exploratory phase, the main propose of phase I was to clear concepts between TAS and FAS and select a suitable FAS measuring method. Focusing on woodchips, different FAS measuring methods were tested and compared in this phase. The specific objectives of phase I were to:

- Collect and characterize woodchips, leaves, and biosolids
- Apply the particle density (PD) method, test methods for the examination of composting and compost (TMECC): 03.01 A & B, gas pyconometer (GP) method, and maximum moisture content (MC^{max}) method to a broad range of materials (materials tested for each method varied as the four methods were tested at different times and some materials were run out).
- Compare all methods based on the results of the same material (3.18 – 9.53 mm woodchips) and select an optimum method for measuring FAS.

3.1.1 Collection and Preparation of Raw Materials

Woodchips

Two kinds of woodchips: full-scale fine and sieved full-scale fine were prepared in this phase. In addition, data of full-scale coarse and lab-scale woodchips were also cited for reference. All the four kinds of woodchips were chipped from the same wood waste of Pa'Dor Manufacturing, a pallet manufacturer in the City of Winnipeg. The waste is off-cuts from the manufacturing process and consists mainly of green, untreated poplar wood (*populus balsamifera*; Little, 1971) which is normally disposed of at a sanitary landfill. In order to identify the four kinds of woodchips conveniently, the definitions of the woodchips as well as their preparation were summarized in Table 3-1.

Table 3-1 Definitions and preparations of different woodchips

Definitions	Preparations
Lab-scale woodchips	Prepared from wood waste using a small shredder (Crary BearCat Limited, model 70530, 3HP) in the laboratory.
Full-scale coarse woodchips	Prepared from material received from the pallet manufacturer using a Maxigrind 460 shear shredder (Rexworks, Milwaukee Wisconsin) equipped with a 50.8 mm (2 inch) screen.
Full-scale fine woodchips	Prepared from full-scale coarse woodchips using an EC 156 Rotochopper (Coon Valley, Wisconsin) equipped with a 31.8 mm (1.25 inch) screen.
Sieved full-scale fine woodchips (3.18 - 9.53 mm)	Screened from full-scale fine woodchips by passing (9.53 mm (3/8 inch) screen and retaining on 3.18 mm (1/8 inch) screen

Leaves

Leaves were collected from the "Leaf It with Us" composting depository located at the Brady Road Landfill outside of Winnipeg. Bagged leaves at this site were stacked in windrows of approximately 40-meter in length and 3-meter high. Composite samples were gathered using Method 02.01-B Selection of Sample Locations for Windrows and Piles (USCC, 1997). Four 68 L bins were used to store the leaves.

Biosolids

Municipal dewatered sludge or biosolids were obtained from the North End Water Pollution Control Center, Winnipeg. The biosolids were stored in a sealed 20 L container and stored at 4°C.

Sampling Methods

Generally, spatial randomization is used to collect samples from windrows or material piles (USCC, 2001). The first step of the spatial randomization procedure was to determine or estimate the number of pace lengths around the perimeter of the windrow or pile being sampled. Twelve points were randomly selected and a 10L sample was collected from the top, middle, and bottom of the pile, separately. The samples were combined to form a gross sample. Sub-samples of approximately 250mL were then randomly selected from the gross sample.

Analysis aliquots were obtained from the sub-samples using the quartering method.

3.1.2 Material Characterization

Materials used in this phase were mainly woodchips, but leaves were also used for comparison. Woodchips were analyzed for MC, BD, and PD. Characterization of leaves was cited from Gagne (2001).

Moisture Content

Moisture contents (MC) were calculated from the total solids (TS) results of samples. TS test was guided by standard method 2540-B (APHA 1995). The calculation was expressed as the following:

$$\% MC = 1 - \% TS \quad [3-1]$$

Bulk Density

Compressive cell (refer to Figure 2-5; P. 23) was used to determine unloaded bulk densities of samples and the calculation was expressed as following:

$$BD = \frac{(W_{cs} - W_c)}{V_s} \quad [3-2]$$

Where BD is the bulk density of sample (g cm^{-3}); W_{cs} is the weight of cell plus sample (g); W_c is the weight of cell (g); V_s is the volume of sample (cm^3). The procedure of wet bulk density measurement of woodchips, leaves, as well as their mixture with biosolids is presented in Table 3-2.

Table 3-2 Procedure of wet bulk density determination

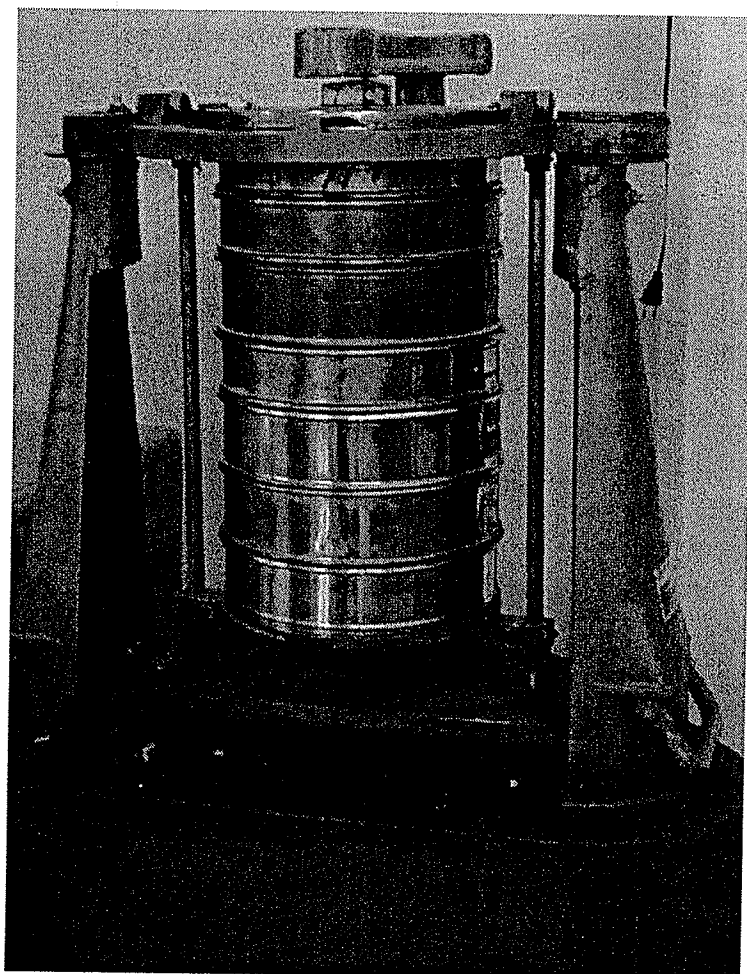
Steps	Descriptions
1.	Weigh an empty cell with known volume.
2.	Add sample into the cell to approximate 1/3 full. Allow the cell containing sample to fall freely onto rubber mat once from height of 15 cm (USCC, 2001).
3.	Repeat step 2 two more times until the cell is almost full. Top off the cell with some more sample.
4.	Record the weight of filled cell. Mass of the sample is the weight difference between the filled cell and the empty cell.
5.	Divide sample mass by the volume of sample to calculate the wet bulk density.

Particle Size Analysis

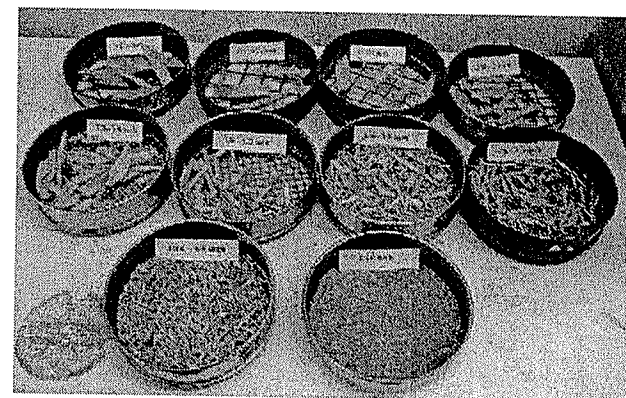
Particle size analysis was performed for both full-scale fine and coarse woodchips, and the analysis equipment is presented in Figure 3-1. The equipment included a series of US standard sieves with different opening sizes: 0.79 mm (1/32"), 1.59 mm (1/16"), 3.18 mm (1/8"), 6.35 mm (1/4"), 12.70 mm (1/2"), 19.05 mm (3/4"), 25.40 mm (1"), 31.75 mm (1.25"), and 38.1 mm (1.5"). A sieve shaker (model CL-430, Soiltest Incorporated, Illinois) was used to perform shaking (Figure 3-1). The procedure of particle size analysis is listed in Table 3-3.

Table 3-3 Procedure of particle size analysis

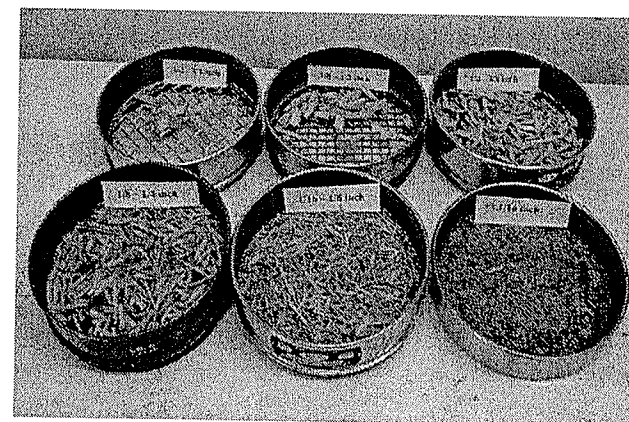
Steps	Descriptions
1.	Weigh each empty sieve and record the readings.
2.	Assemble cap, sieves, and pan from top to bottom (larger size sieves on top) and put them in the sieve shaker; adjust the bottom plate of the shaker to make sure that the sieves and shaker contact tightly
3.	Take the assembled sieves out and fully fill sample into the top sieve.
4.	Turn on the shaker for 10 minutes
5.	Take the assembled sieves out from the shaker; weigh each sieve containing sample
6.	Particle size is expressed as a range; for example, 0.79 mm (1/32") – 1.59 mm (1/16") sample was those passing 1.59 mm (1/16"), and retained on 0.79 mm (1/32") sieve.



(a) Shaking machines and combined standard sieves



(b) Sieved coarse woodchips



(c) Sieved fine woodchips

Figure 3-1 Sieve shaker, sieves and sieved woodchips

Particle Density

Particle density was determined using pycnometer method (Klute et al. 1986). In this method, particle density is calculated from two measured quantities, i.e. mass of sample and its volume. The mass is determined by weighing, and the volume is calculated from the mass and density of water displaced by the sample. The calculation was expressed as the following;

$$PD = \frac{\rho_w \cdot (W_s - W_p)}{(W_s - W_p) - (W_{sw} - W_w)} \quad [3-3]$$

where PD is the particle density (g cm^{-3}); ρ_w is the density of water (1 g cm^{-3}); W_s is the weight of pycnometer plus sample (g); W_p is the weight of pycnometer filled with air (g); W_{sw} is the weight of pycnometer filled with sample and water (g); W_w is the weight of pycnometer filled with water at temperature observed (g). The procedure of particle density measurement is presented in Table 3-4.

Table 3-4 Procedure of particle density determination modified
for compost materials

Steps	Descriptions
1.	Weigh a clean and dry 1000 mL flask.
2.	Fill flask with distilled water (pre-boiled) to 1000 mL mark line and weigh the flask with water; Remove water and dry the flask in 103 °C oven.
3.	Add approximate 50 g sample to the cooled flask, ensuring that the outside of the flask is clean after addition.
4.	Weigh the flask with compost
5.	Fill the flask about half full with distilled water. Boil the mixture on electric heater until most samples settled down.
6.	Cool the flask to room temperature and add distilled water (pre-boiled) to the 1000 mL mark line; dry the outside of the flask.
7.	Weigh the flask with sample and water; Record the room temperature.

3.1.3 Preparation of Feedstock

Sieved Full-scale Fine Woodchips (3.18 – 9.53 mm)

In this experiment, model 1350 multivolume pycnometer was used as a FAS measurement apparatus. Due to its limited chamber volume, full-scale fine woodchips (0 – 19.5 mm) was too large to fit in. For this reason, smaller size of

woodchips (3.18 – 9.53 mm) was selected from the full-scale fine woodchips (0 – 19.05 mm). A combined sieve (9.53 mm on top and 3.18 mm on bottom) was used to select the woodchips, and the procedure was the same as that of particle size analysis presented in Section 3.1.2.

Composting Mixtures

Mixtures of woodchips/biosolids and leaves/biosolids were used in experiment of phase I. All mixtures were made to target moisture content (MC) of 55 % and the amount of amendments and biosolids were calculated using the following equation:

$$MC_m \bullet m_m = MC_b \bullet m_b + MC_{bs} \bullet m_{bs} \quad [3-4]$$

Where MC_m is the target moisture content of the feedstock mixture (0.55); m_m , m_b , and m_{bs} are the wet mass (g) of feedstock mixture, bulking agents, and biosolids, respectively; and MC_b and MC_{bs} are the fractional moisture contents of the bulking agents and biosolids, respectively. All mixtures were mixed using a Hobart model AS-200-FDT mixer (Hobart Corporation, Troy Ohio).

3.1.4 FAS Determination Method

In this phase, four FAS determination methods, including particle density (PD) method, TMECC method 03.01 A & B, maximum moisture content (MC^{max}) method, and gas pycnometer (GP) method, were used for comparing the results.

Particle Density (PD) Method

As a traditional FAS determination method, PD method was used by many researchers in composting engineering field (Das and Keener, 1997; Chen, 1998; Leonard and Ramer, 1993). The operation procedure of particle density method can be borrowed from the procedures of bulk density and particle density presented in section 3.1.2. FAS was then calculated according to equation 2.6, 2.7 and 2.8 presented in section 2.3.2.1.

TMECC Method 03.01 A & B

The theory of this method is water-saturation. Sample is saturated with water and excess water is drained, and BD, porosity, and FAS of sample are measured by measuring the sample volume changes, water retained, and water drained.

BD was calculated using the following equation:

$$BD = ODW_{1800} / V_{1800} \quad [3-5]$$

where BD is the bulk density of sample (g cm^{-3}); ODW_{1800} is the oven dry weight of 1800 mL sample (g); V_{1800} is the initial volume of compost in the 1800 cm^3 cylinder (cm^3). The pore space was calculated as the following:

$$PS, \% = \frac{(W_{1800WS, no\ drain} - ODW_{1800})}{V_{1800}} \times 100 \quad [3-6]$$

where PS , % is the pore space (fractional, $\text{cm}^3\text{cm}^{-3}$); $W_{1800WS, no\ drain}$ is the weight of water-saturated sample before 4-hour draining (g). The FAS was calculated as the following:

$$FAS, \% = \frac{(W_{1800WS, no\ drain} - W_{1800WS, after\ drain})}{V_{1800}} \times 100 \quad [3-7]$$

where FAS , % is the free air space (fractional, $\text{cm}^3\text{cm}^{-3}$); $W_{1800WS, after\ drain}$ is the weight of water-saturated sample after 4-hour draining (g). For the detailed procedure of TMECC method 03.01 A & B, please refer to the original document (USCC, 2001).

Gas Pycnometer Method

The gas pycnometer was originally designed to measure air-filled porosity of unsaturated soils (Kummer and Cooper, 1945; Mohsenin, 1986). This method is based on Boyle's gas law and it has been applied by many investigators (Kummer and Cooper, 1945; Mohsenin, 1986; and Visvanathan, 1996). More recently, Modified gas pycnometers were reported to successfully measure FAS of composting materials (Oppenheimer, 1997; Agnew and Leonard 2002).

This method measured the skeletal volume of sample by measuring the pressure change of working gas before and after diffusion in the sample. The skeletal volume was calculated as the following:

$$V_s = V_c - \frac{V_E}{P_1/P_2 - 1} \quad [3-8]$$

where V_s is the skeletal volume of sample (cm^3); V_c is the volume of cell used to hold the sample (cm^3); V_E is the expansion volume added when the sample is under test (cm^3); P_1 is the pressure of working gas before it diffuse into the sample (psig); P_2 is the pressure of working gas after it diffused into the sample (psig). The FAS was calculated as:

$$FAS = \frac{V_B - V_s}{V_B} \quad [3-9]$$

where V_B is the bulk volume of sample (cm^3). For the detailed procedure of gas pycnometer method, please refer to the operation manual of Model 1350 Multivolume Pycnometer (Micromeritics Instrument Company).

Maximum Moisture Content (MC^{max}) Method

As mentioned on the above, particle density method measures the TAS, which includes the inter-particle, i.e. FAS, and internal air space. According to Equation 2-16, FAS equals to TAS subtracting micro-pores. Therefore, FAS would be determined if volume of micro-pores were measured. In order to measure the maximum MC inside the sample, the water between particles has to be removed after the sample is water-saturated in the process of particle density method. In the experiment of phase I, paper towel was adapted to adsorb the water between particles, and this method was called “maximum moisture content (MC^{max}) method”. The suitability test and standard procedure development of this method was then conducted in phase II.

Referring to the mathematical models developed by Eftoda and McCartney (2002), the procedures for the FAS determination were the following (the detailed procedure was presented in Table 3-5):

- 1) Mass of water in the sample

$$m_w = m_t \cdot MC \quad [3-10]$$

where m_w is the mass of water in the sample (g); m_t is the mass of woodchips (g); and MC is the moisture content of the woodchips (fractional).

- 2) Maximum mass of water in water-saturated sample (m_w^{\max})

$$m_w^{\max} = \left[\frac{m_t(1 - MC)}{1 - MC^{\max}} \right] \cdot MC^{\max} \quad [3-11]$$

Where MC^{\max} is the maximum moisture content of the water-saturated sample (fractional).

- 3) The volume of air in the micro-pores of the sample (V_a)

$$V_a = \frac{m_w^{\max} - m_w}{\rho_w} \quad [3-12]$$

- 4) FAS was calculated as:

$$FAS = TAS - \frac{V_a}{V_t} \quad [3-13]$$

where V_t is the total volume of the sample (cm^3).

Table 3-5 Procedure of MC^{max} method for bulking agents ^[1]

Step	Description
1.	Perform particle density analysis (refer to Table 3-2)
2.	Use a fine sieve (0.79 mm) to drain off water from the water-saturated sample
3.	Place the sample on multi layer paper towels on the table; make the sample a uniform thin layer; use paper towel to gently blot sample to adsorb the water outside of sample particles until no water glistening on the surface of sample; change paper towel once it is wetted.
4.	Transfer blotted samples (triplicate) into crucibles and dry in a 103 °C oven for 24 hrs to determine MC ^{max} .
5.	Perform calculations to determine FAS using the MC ^{max} and other results determined before.

^[1] This procedure was only used in phase I and a standard procedure for MC^{max} method was developed and described in section 4.2.1.

3.2 Objectives and Techniques of Experiment Phase II

The objective of experiment phase II was to develop a standard procedure for the maximum moisture content (MC^{max}) according to the standard method 1040: method development and validation (APHA 1995). FAS of compost materials can be calculated using MC^{max} and some equations known before. Therefore,

completion of the standard procedure development of MC^{max} method was the key step to accomplish the development of a standard method.

3.2.1 Material Preparations

Material used in this phase included full-scale fine woodchips, straw, and wood shavings.

Woodchips

Woodchips used in this phase were the full-scale fine woodchips (0 - 19.05 mm) used in phase I. However, in order to investigate the reliability and applicability of MC^{max} method, two kinds of sieved woodchips were used: 1) 0.79 - 19.05 mm and 2) 6.35 - 12.7 mm.

The size selection was performed by using standard sieves as well as their combinations. Woodchips with size of 0.79 - 19.05 mm were those full-scale fine woodchips retained on 0.79 mm sieve, and woodchips with size of 6.35 - 12.7 mm were those passed 12.7 mm sieve but retained on 6.35 mm sieve. The procedure of size selection was the same as that of particle size analysis presented in Section 3.1.2.

Straw

The straw was collected from the Dow Bioproducts Inc. strawboard manufacturing facility located near Elie, Manitoba. The major straw variety

present in the Dow Bioproducts stack yard is AC Barrie wheat straw, a predominant variety of wheat farmed in the 50-80 km feedstock collection radius of the strawboard manufacturing facility. Although 15 varieties of straw are present in the stack yard, over 75% of the straw stock is of the AC Barrie variety (Nechwediuk, 2002). The straw was obtained from 1m x 1m x 2m rectangular bales in the Dow stack yard.

The bales were sampled on April 10, 2002 and shredded by an on-site tub grinder (model: H1100E, Haybuster). The shredded samples were then put in a large white bag and transported back to the U of M where they were stored in an outside storage shed.

Wood shavings

Wood shavings used in this phase was Living World[®] pine shavings (14-L; made in Holland).

3.2.2 Experimental method and apparatus

All materials used in phase II were analyzed for moisture content (MC), bulk density (BD), particle density (PD), and maximum moisture content (MC^{max}). Experimental methods and apparatus for MC, BD, PD, and MC^{max} were described

in section 3.1.2. For MC^{max} determination, a standard procedure of MC^{max} method was developed as part of results presented in section 4.1.2.

3.3 Objectives and Techniques of Experiment Phase III

The objective of experiment phase III was to apply the developed MC^{max} method to compost mixtures, improve the current FAS method, and complete the whole standard FAS method development. Specifically, the objectives of phase III include the following:

- Apply the MC^{max} method to compost mixture and quantify the validations
- Determine the accuracy of PD measuring method by applying the method to different size of woodchips with different boiling time and MC
- Combine the MC^{max} method with Haug's conceptual model to calculate the FAS of compost mixtures
- Complete the development of standard FAS measurement method

3.3.1 Materials preparation

Materials used in this phase were the same materials collected in phase I: 1) full-scale fine woodchips (0 - 19.05 mm); 2) straw; 3) biosolids

Woodchips

In order to investigate the effects of particle size on FAS measurement, both original (0 -19.05 mm) and sieved full-scale fine woodchips (6.35 -12.7 mm) were adopted in this experiment. The procedure for size selection referred to that of particle size analysis presented in Section 3.1.2.

Compost Mixtures

Compost mixtures were made by biosolids and bulking agents with a target MC of 55 % using Equation 3-4. Three kinds of compost mixtures were prepared in this phase: 1) biosolids with woodchips (0 – 19.05 mm); 2) biosolids with woodchips (6.35 – 12.7 mm); and 3) biosolids with straw. The procedure of compost mixture preparation was described in Section 3.1.3.

3.3.2 Experimental method and apparatus

All materials were analyzed for moisture content (MC), bulk density (BD), particle density, and maximum moisture content (MC^{max}). The experimental methods and apparatus for these parameters were described in section 3.1.2.

Except the regular analysis methods, a conceptual model (Haug, 1993) for estimating volumetric mixing ratio of composting mixtures was cited here to determine FAS of composting mixtures. This model was established on the theory of moisture transfer between wet substrate and bulking agents. According to Haug

(1993), six assumptions were made for this model. All of them were met by composting mixtures used in this experiment, and the detailed analysis was conducted in section 4.3.2. Haug's conceptual model for estimating ratio of substrate to bulking agents has two styles: 1) bulking agent limited and 2) substrate limited. The former style was adopted in this experiment because all bulking agents used in the mixtures were not saturated (i.e. not reach the maximum MC). The conception model (bulking agents limited) was expressed as the following:

$$\frac{1}{R_{bs}} = \left(\frac{BD_b}{\rho_w}\right)\left(\frac{S_b}{S_{bm}^m} - 1\right) + R_{mb}(1 - FAS_m) - (1 - FAS_b) \quad [3-14]$$

And it was rearranged as the following for FAS determination:

$$FAS_m = 1 - \frac{1}{R_{mb}} \left[\frac{1}{R_{bs}} + (1 - FAS_b) - \left(\frac{BD_b}{\rho_w}\right)\left(\frac{S_b}{S_{bm}^m} - 1\right) \right] \quad [3-15]$$

Where FAS_m is the FAS of compost mixture ($\text{cm}^3\text{cm}^{-3}$); FAS_b is the FAS of bulking agents ($\text{cm}^3\text{cm}^{-3}$); R_{bs} is the volumetric ratio of bulking agents to substrate (biosolids); R_{mb} is the volumetric ratio of compost mixture to bulking agents; BD_b is the wet bulk density of bulking agent (gcm^{-3}); ρ_w is the density of water (gcm^{-3}); S_b is the solid content of bulking agents (fractional); and s_{bm}^m is the minimum solid content of bulking agents after the mixing with substrate (fractional).

According to the rearranged Haug's conceptual model, FAS of composting mixture can be worked out if all parameters in equation 3-14 are known. From equation 3-14, obviously, R_{bs} , R_{mb} , and BD_b can be measured or calculated easily using

procedure for bulk density. ρ_w can be assumed as 1.0 gcm^{-3} without considering temperature effects. S_b is determined using APHA method 2540B. FAS_b can be measured by MC^{max} method. As to S_{bm}^m , some assumptions are required to calculate it.

In this experiment, substrate (MC_{sm}), bulking agents (MC_{bm}), and mixture (MC_m) were assumed to have the same moisture content after mixing. In the above model (bulking agent limited), the minimum solid content of bulking agents (S_{bm}^m) is equal to the solid content of bulking agent in mixture (S_{bm}) (Haug, 1993). Combined with the conditions in this experiment, S_{bm}^m can be calculated as:

$$S_{bm}^m = S_{bm} = 1 - MC_{bm} = 1 - MC_m = S_m \quad [3-16]$$

Where S_{bm} is the solid content of bulking agents in mixture after moisture absorption; S_m is the solid content of mixture.

Therefore, Haug's conceptual model applied in this experiment can be rearranged as the following:

$$FAS_m = 1 - \frac{1}{R_{mb}} \left[\frac{1}{R_{bs}} + (1 - FAS_b) - \left(\frac{BD_b}{\rho_w} \right) \left(\frac{S_b}{S_m} - 1 \right) \right] \quad [3-17]$$

3.3.3 Statistical Analysis Method

Statistical analysis was applied to determine whether there is significant difference existing for experimental results caused by different conditions: materials, methods, and operators. If significant statistical difference existed, the condition caused that was considered important for a method. SigmaStat® software (version 2.0) was used to perform t-test for two groups of results and one way analysis of variance (one way ANOVA) for three or more groups of results.

Chapter 4

Results and Discussions

The experimental program consisted of three Phases. Phase I focused on comparison of currently available methods for measuring FAS. Phase II was to develop standard procedure of MC^{max} method. Phase III analyzed the limitation of MC^{max} method and made improvement by proposing a FAS numerical model. All experimental data collected are presented in Appendices. Summarized results and discussion are presented in this chapter.

4.1 Results and Discussions of Phase I

Phase I investigated the suitability and effectiveness of four different methods: PD method, TMECC method, GP method, and MC^{max} method. Different sizes of woodchips were used to test each method, but only the FAS results from woodchips with size of 3.18 – 9.53 mm were used for the comparison of four methods and selection of an acceptable method to be standardized.

4.1.1 Characterization of Raw Materials

Raw Materials used in this Phase were: 1) woodchips (different sizes); 2) leaves; and 3) biosolids. Woodchips used in this Phase mainly included lab-scale and full-scale fine woodchips, and they were characterized for size, MC, BD, and PD. Also, characterization result of full-scale coarse woodchips was cited for comparison. Relationship among the three kinds of woodchips was expressed in Figure 4-1. Also, characterization result of leaves was cited from Gagne (2001). Biosolids was only characterized for MC. The characterization results of all raw materials are summarized in Table 4-1.

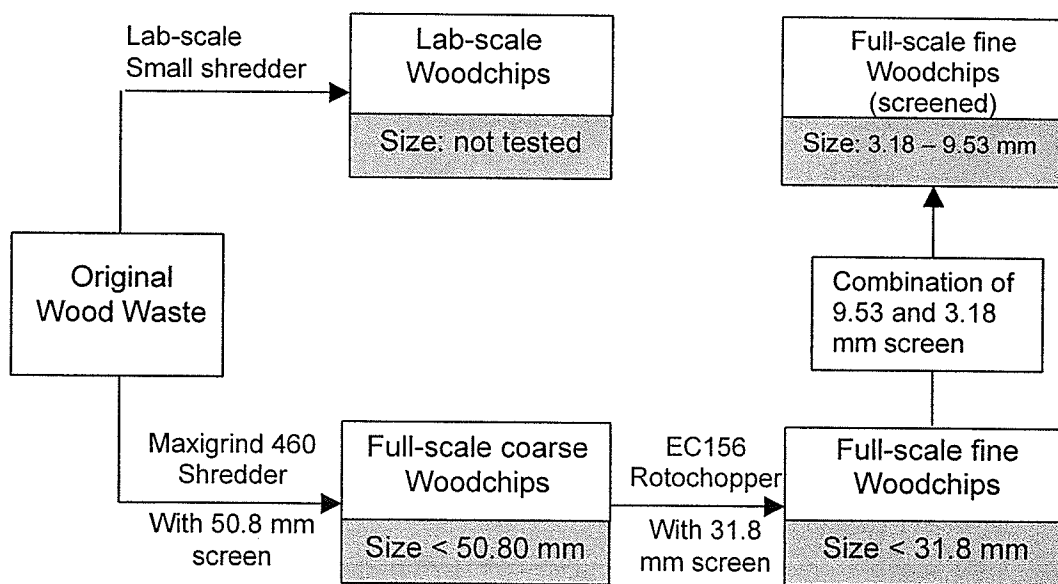


Figure 4-1 Relationship among woodchips originated from the same wood waste

From Table 4-1, particle size was dramatically different between full-scale coarse and fine woodchips. Referring to the particle size analysis (Table A-4-1 and A-4-2; P. 111), 28 % (weight percentage) particles in full-scale coarse woodchips had a size larger than 19.05 mm, the high limit of particle size for full-scale fine woodchips.

Table 4-1 Characterization of Raw Materials ^[1]

Woodchips	Size (mm)	MC (%)	BD (g cm ⁻³)		PD (g/cm ³)
			Wet	Dry	
Full-scale fine	0 – 19.05	31.51	0.2365	0.1260	1.66
Sieved full-scale fine	3.18 - 9.53	38.47	0.1588	0.0978	1.54
Full-scale coarse ^[2]	0 – 50.80	21.36	0.1583	0.1245	1.74
Lab-scale ^[3]	Unknown	41.03	0.2295	0.1353	1.80
Leaves ^[3]	Unknown	38.68	0.1532	0.0940	1.89
Biosolids	Unknown	71.95	---	---	---

^[1] Two samples used for particle size and BD test; three samples used for MC and PD test; individual %RSD of different materials for each parameters were presented in appendix A (P.107-113)

^[2] Data from Eftoda and McCartney (2002)

^[3] Data from Gagne (2001)

MC varied among different raw materials. Biosolids was known as high MC and zero FAS material and its MC in this experiment was as high as 71.95 %. For woodchips originating from same wood waste, full-scale coarse woodchips had lowest MC because it was dried on the field in summer time before collection. Full-scale fine woodchips was collected in winter time and it absorbed some

waters from the snow covering on it, so its MC was relatively higher than that of full-scale coarse woodchips. Lab-scale woodchips were chipped from fresh wood waste and thus contained moisture as over 40 %.

Wet BD of four different woodchips ranged from 0.1583 to 0.2365 g cm⁻³. As mentioned in Chapter 1, wet BD was affected by both MC and particle size, therefore the dry BD would be much more convenient to figure out the effects of particle size on BD variation. Generally, large size particles provide the structure support and porosity but fine ones always take up inter-particle voids. Therefore, for a given volume, dry BD of large size woodchips is always lower than that of small ones. For this reason, full-scale coarse woodchips had lower dry BD than that of full-scale fine woodchips. Also, sieved full-scale fine woodchips has a much lower dry BD than that of full-scale fine woodchips because the sieved one did not have any minute particles occupying their inter-particle voids.

Generally, particle density of a certain material does not change with particle size and MC. In this experiment, the mean values of particle densities varied among different sizes of woodchips, but no significant statistical difference was found among them (statistical report D-3; P.147).

4.1.2 Individual Results of four FAS Methods

4.1.2.1 FAS by Particle Density (PD) Method

PD method was successfully used to measure FAS of different feedstock, including woodchips, leaves, and mixtures of woodchips/biosolids and leaves/biosolids (Chen, 1998; Gagne 2001). As mentioned earlier, “FAS” measured by PD method actually includes intra- and inter-particle voids, which is obviously greater than the FAS (inter-particle voids) defined in this paper. The analysis of how FAS measured by PD method deviated from the true value was conducted in section 4.1.3. In this experiment, PD method was applied to different sizes of woodchips originated from Poplar (*populus balsamifera*) wood waste to investigate its suitability and accuracy. As mentioned earlier, FAS is related to porosity and moisture content (MC), so both porosity and FAS results were presented in Table 4-2.

Table 4-2 Porosity and FAS of woodchips measured by PD method

Woodchips	Number of Samples	Porosity (%)	FAS (%)	RSD (for FAS) (%)
Full-scale fine	3	90.23	82.78	0.35
Sieved full-scale fine	3	94.11	88.00	0.20
Full-scale coarse ^[1]	2	92.81	89.43	0.78
Lab-scale ^[2]	4	92.30	82.91	1.76

^[1] Results were calculated using data from Eftoda (2002)

^[2] Results were calculated using data from Gagne (2001)

From Table 4-2, for different woodchips, porosities ranged from 90 – 94 % and FAS ranged from 83 – 89 %. The differences between porosities and FAS were caused by their MC. For example, full-scale coarse and lab-scale woodchips has almost the same porosities but their FAS were dramatically different (statistical report D-4; P.148) due to their different MC (21.36 % and 41.03 %). Focused on woodchips from the full-scale project (i.e. full-scale fine, sieved full-scale fine and full-scale coarse woodchips), two phenomena were obvious: 1) full-scale coarse woodchips had the highest FAS and 2) screened full-scale fine woodchips had higher FAS than that of unscreened ones. These two phenomena are related to two important factors: moisture content and particle size. According to Equation 2-10, FAS decreases with volumetric water content. In addition, large size particles are able to provide good porous structure so that they will have more inter-particle voids. In this experiment, full-scale coarse woodchips had the highest FAS among all woodchips just because they had lowest MC and largest particle size. As to sieved and un-sieved full-scale fine woodchips, their MC were 38.47 % (higher FAS) and 31.51 %, respectively. Although sieved full-scale fine woodchips had higher MC, its FAS was still higher (88.00 %) than that of unsieved ones (82.78 %). It was probably that sieved full-scale fine woodchips did not contain any minute particles occupying inter-particle voids.

According to APHA (1995), analytical results with low relative standard deviation (RSD) indicate high precision of a method. So, a $RSD < 3\%$ is suggested in this experiment for a precise method. In this experiment, RSD for all FAS of different

woodchips were lower than 3 % and it indicate that PD method is precise enough. However, high precision does not mean accurate. As mentioned, FAS measured by PD method is not only inter-particle but also intra-particle voids, which deviate from the definition of FAS: inter-particle voids. Therefore, PD method has high bias. According to Figure 4-2, the definition by APHA (1995), an accurate method must have qualified data with high precision and low bias. For this reason, PD method can not be taken as an accurate method for FAS measurement, but can be considered as an accurate method for TAS measurement.

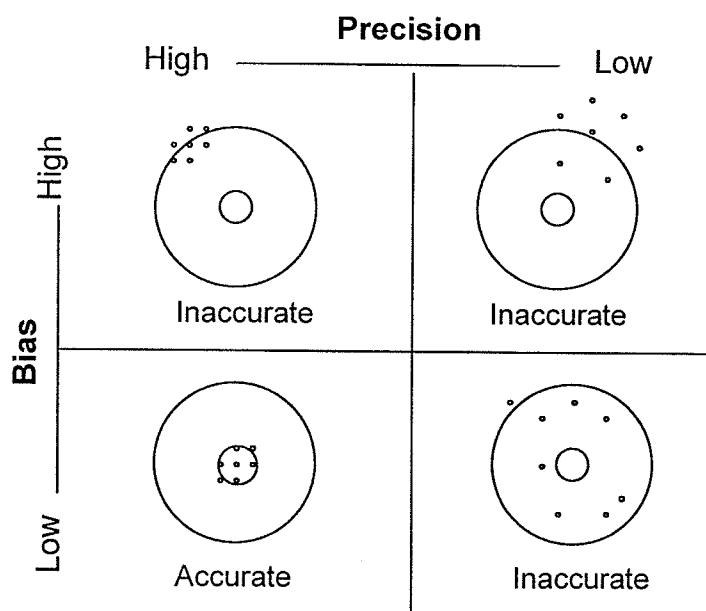


Figure 4-2 Definition of Accuracy (APHA, 1995)

4.1.2.2 TMECC Method 03.01 A & B

Reported by USCC (2001), TMECC method 03.01 A & B were able to measure porosity and FAS of composting materials. Referred to the procedure, FAS by TMECC method represents inter-particle voids in a sample. Theoretically, FAS by TMECC method should be very close to the true value. In this experiment, TMECC method was applied to four kinds of materials: woodchips (3.18 - 9.53 mm), leaves (unknown size), and their mixtures with biosolids: biosolids/woodchips and biosolids/leaves. Results by TMECC method were presented in Table 4-3, and FAS by PD method were cited for comparison purpose. In addition, porosities by both methods were also quoted for investigating effectiveness of TMECC method.

Table 4-3 Comparison of Porosity and FAS by TMECC and PD method

Materials	Number of Samples		Porosity (%)		FAS (%)		RSD for FAS (%)	
	TMECC	PD	TMECC	PD	TMECC	PD	TMECC	PD
Sieved full-scale fine Woodchips	3	3	73.02	94.11	43.21	88.00	3.82	0.20
Leaves ^[1]	2	3	78.13	95.03	47.19	89.10	0.43	0.45
Biosolids/woodchips ^[2]	2	3	42.22	89.59	13.13	70.72	11.64	0.16
Biosolids/Leaves ^[1]	2	3	70.32	91.20	26.11	74.96	3.81	0.07

^[1] Results by PD method are cited or calculated from Gagne (2001)

^[2] Woodchips in the mixture is sieved full-scale fine woodchips (3.18 – 9.53 mm)

As shown in Table 4-3, both porosities and FAS measured by TMECC method were much lower than those by PD method, and it was confirmed by the

statistically significant differences between them (Appendix D-5 and D-6; P.149-150). Specially, FAS of bulking agents (woodchips and leaves) were extremely low compared with those of mixtures (biosolids/woodchips and biosolids/Leaves). Although these differences had inseparable relations to properties of samples, the method itself was the main factor. Two phenomena related to the defects of the method were observed during the experiment: 1) insufficient water saturation of samples and 2) poor drainage. Referring to the procedure of TMECC method 03.01 A & B, the first phenomenon lower the weight of water-saturated sample ($W_{1800WS, no\ drain}$) and the second one increased the weight of water-saturated sample after 4-hour draining ($W_{1800WS, after\ drain}$). According to Equation 3-6 and 3-7, it is clear that both phenomena contributed for lower porosity and FAS measurements.

In this experiment, porosities measured by TMECC method ranged from 42 % to 75 %, while those by traditional PD method were high as over 90 %. This significant difference (Appendix D-5; P.149) attributed to the extent of water saturation of samples and insufficient water saturation was the direct reason for low porosities by TMECC method. It was observed during the process of TMECC method that woodchips or leaves were floating in beaker after initial water addition. After 3 times repeat (approximate 90 minutes) of water adding and draining, all leaves settled down but a few larger size woodchips still kept on floating. This phenomenon indicated that the water-saturation of woodchips and

leaves were not enough. Floating did not happen to mixtures because their densities were heavier than bulking agents alone. However, it can be deduced that the water-saturation would not be complete because biosolids attached on the surface of bulking agents make the water absorption more difficult. The non-saturation status meant that air spaces inside sample were not replaced by water completely, so the porosity values measured were much lower than those measured by PD method.

As to the dramatic differences of FAS between bulking agents and mixtures, they were affected by two factors: 1) the property of biosolids and 2) poor drainage during the experiment. As mentioned, biosolids have especially high moisture content and extremely low FAS (near to zero). When biosolids were mixed with bulking agents, fine particles from biosolids occupied inter-particle voids of bulking agents and lower FAS of the mixture. The lower FAS of mixture affected by biosolids were shown for both PD and TMECC method. However, the effect from biosolids was only the auxiliary factor for extremely low FAS of mixtures by TMECC method and defects of the method itself were the crucial ones. Poor drainage caused by fine particles was observed during the application of TMECC method and it was responsible for the low FAS. Poor drainage made water retain among sample particles and increased the weight of water-saturated compost after draining ($W_{1800WS, \text{ after draining}}$) than it should be. Consequently, higher $W_{1800WS, \text{ after draining}}$ finally lowered the FAS of sample according to Equation 3-7 (P.45).

Blocking was very serious for the mixtures because particles of biosolids were much finer than those of woodchips and leaves, and it was confirmed by their extremely low FAS (13.13 % for biosolids/woodchips and 26.11 % for biosolids/leaves).

In addition, all RSD of FAS of different materials by TMECC method were higher than 3% and it indicated a lower acceptable precision. Although this method has a theoretical ability to measure inter-particle voids (refer to section 2.3.2.3), its ineffective procedures produced unacceptable low results. Therefore, this method was considered to have high bias. According to the accuracy definition by APHA (1995), TMECC method was not accurate.

Based on the above analysis, FAS by TMECC method was not reliable and accurate because of the defects of the method itself. Further research and improvement are necessary to make it accurate and effective for composting materials.

4.1.2.3 Gas Pycnometer (GP) Method

Modified gas pycnometer was reported to successfully measure FAS (or air-filled porosity) of composting materials (Oppenheimer, 1997; Agnew and Leonard 2002). In addition, Webb (2001) pointed out that GP method measured air-filled porosity that includes inter-particle voids and open pores (part of intra-particle

voids). Consequently, FAS measured by this method is higher than the true FAS (inter-particle voids).

Commercial gas pycnometer was used in this experiment to investigate its precision of measuring FAS. As suggested by the manufacturer, sample must be dry in order to obtain accurate results because of the interference from the water vapor. In practice, most composting materials are not totally dry. To investigate the interference from water, GP method was applied to both oven-dried and as-received woodchips. Triplicate tests were completed for each sample and results are summarized in Table 4-4.

Table 4-4 FAS of sieved full-scale fine woodchips (3.18 - 9.53 mm)
measured by GP method

Sample	Number of Sample	MC (%)	FAS (%)	RSD ^[1] (%)
Oven-dried	3	0	92.78	0.11
As-received	3	38.47	83.07	0.43

^[1] RSD is for FAS

First, as shown in Table 4-4, FAS of oven-dried sample were higher than that of as-received sample and the difference between them was approximately 10 %. For the as-received sample, obviously the moisture content occupies some open pores inside sample and lowers its FAS measurement. Referring to Table A-6-2 (P.114), the volumetric water content in as-received sample was 6.11 %, which exist in

both open and closed pores. Assuming all water content was in open-pores, the theoretical FAS value of as-received sample by GP method should be 86.67 % that equals to FAS of oven-dried sample (92.78 %) subtracting the volumetric water content (6.11 %). However, FAS of as-received sample measured in this experiment was only 83.07 %. The possible reason for this difference (3.6 %) was the interference from water vapor. Under high pressure, moisture content inside as-received sample turned into water vapor and made the final pressure (P_2) higher. According to Equation 3-8 and 3-9, higher final pressure (P_2) will cause higher skeletal sample volume (V_s) and hence lower the FAS. Therefore, moisture content in sample actually decreased its FAS. Although the moisture interference on FAS was mentioned by the manufacturer of gas pycnometer (Micromeritics Instrument Company) and reflected in this experiment, it was not reported by other researchers using gas pycnometer (Oppenheimer, 1997; Agnew and Leonard, 2002).

Second, GP method can be considered as a precise and quick method. As shown in Table 4-3, the RSD of both oven-dried and as-received samples were very low, and it indicated that GP method is a precise method. Besides Also, GP method is a quick method because it only takes 30 minutes to complete a test run compared with about 5 hours required by PD method. However, Webb (2001) pointed out that GP method actually measures inter-particle voids and open pores, which

deviate from the definition of FAS (inter-particle voids only). Therefore, GP method can not be considered as an accurate method for FAS measurement.

Last, two limitations of GP method were noticed during the experiment: 1) the capacity of the commercial gas pycnometer and 2) the dry material requirement. The testing chamber of the commercial gas pycnometer (model 1305) only has a maximum capacity of 150 cm³ (approximate size: height of 7.62 cm and diameter of 5.08 cm) and it was not able to hold composting feedstock with large size of bulking agents. Fortunately, this limitation was already overcome and modified commercial gas pycnometer with enlarged volume were developed by others (Oppenheimer, 1997; Agnew and Leonard, 2002). As to the limitation of dry sample requirement, it was shown as an important factor in this experiment because the interference from water vapor was 3.6 %. Due to all composting materials are not totally dry, GP method is not satisfactory and needs improving for wide applications.

Overall, GP method is precise and quick, but it is considered inaccurate because its measurement includes the volume of open pores. Although modified commercial gas pycnometer was developed recently, the interference from moisture content was not reported. To make GP method accurate, further research is necessary to figure out the deviation of FAS caused by open pores and MC.

4.1.2.4 Maximum Moisture Content (MC^{max}) Method

MC^{max} method was initially proposed by Eftoda and McCartney (2002) when the traditional FAS determination method, i.e. PD method, was found to determine the TAS (inter- and intra-particle voids) not FAS (inter-particle voids). As described in MC^{max} method, sample is completely saturated by boiled water so that maximum MC is able to be determined. FAS was then calculated based on known equations (refer to Equation 3-11, 3-12, 3-13, and 3-14). In order to test its stability and precision, MC^{max} method was applied to lab-scale woodchips, leaves (Gagne 2001) and full-scale fine woodchips (3.18 - 9.53 mm). The results are summarized in Table 4-5.

Table 4-5 FAS of different materials determined by MC^{max} method

Sample	Number of Samples	MC (%)	MC^{max} (%)	FAS (%)	RSD ^[1] (%)
Sieved full-scale fine woodchips (3.18-9.53 mm)	3	38.47	64.42	76.42	0.51
Lab-scale Woodchips	3	41.03	67.43 (70.04 ^[2])	64.30 (60.48 ^[2])	0.43 (0.39 ^[2])
Leaves	3	38.68	78.28	61.17	0.66

^[1] RSD is for FAS

^[2] Samples were not blotted by using paper towel

As shown in Table 4-5, FAS of both kinds of woodchips were higher than that of leaves and it was consistent with those results of previous researches completed (Haug, 1993; Chen, 1998). For the lab-scale woodchips, the maximum MC determinations were performed on paper-blotted and un-blotted samples. The results shown that the paper-blotted woodchips had approximate 3 % lower maximum MC and 3 % higher FAS than those of un-blotted woodchips, and it indicated the importance of excess water removing.

In addition, it was observed that all maximum MC were approximately 1.5 – 2 times higher than background MC for all materials. It suggests that bulking agents have a large amount of intra-particle voids, which may affect FAS measurement. If these intra-particle voids were not completely displaced by media (e.g. water), FAS measurement would deviate from the true value.

The main limitation of this method was found to be the interference from fine particles. A few fine particles lost during the paper-blotting operation and it may affect MC^{max} measurement and FAS determination. The loss of fine particles was especially obvious for composting mixture (e.g. biosolids/woodchips and biosolids/leaves) because these samples contained much more fine wet particles. The interference from fine particles was quantified and statistically analyzed in Phase II.

4.1.3 Comparisons of Four Methods

In the previous sections, individual results from different methods were presented and discussed. In order to summarize and compare the four methods across-the-board, it was necessary to systematically analyze the methodological control factors of the four methods.

The method comparison was based on the results from the same size range sample, i.e. woodchips (3.18 - 9.53 mm). To quantitatively analyze the difference among different methods, a hypothesis of true value had to be made. As mentioned previously (Table 2-3, P. 31), FAS by TMECC, MC^{max}, and dry-flow method measures inter-particle voids, which should be mostly close to the true value. As dry-flow method was not adopted and TMECC method showed its ineffectiveness, FAS by MC^{max} method became the only qualified one that close to the true value. Accordingly, it was assumed as the true value.

As shown in Table 4-6, all methods were compared using the following five aspects:

- Precision: the degree of agreement among replicate analysis of a sample (APHA 1995). It was expressed as % RSD.
 - High precision: $RSD < 3\%$
 - Low precision: $RSD > 3\%$

- Bias: consistent deviation of measured FAS from the true FAS (APHA 1995). It is quantified as difference between measured and true value.
- Test duration: time required to complete a whole test
- Cost: cost for apparatus and operation (rough estimation by author)
- Reliability: the suitability and repeatability of a method. Determined by limitation or defects of a method.

Table 4-6 Comprehensive comparisons of four FAS methods

Method	Number of Samples	FAS (%)	RSD (%)	Bias (%)	Test Duration	Cost	Reliability
PD	3	88.0	0.20	+11.58	3-5 hrs	Low	High
TMECC	2	43.21	3.82	-33.21	6-8 hrs	Low	Low
GP	3	83.07	0.43	+6.65	0.5 hrs	high	Medium
MC ^{max}	3	76.42	0.51	0 *	3-5 hrs	low	Medium

* Theoretical bias because of the absence of standard value

From Table 4-6, except TMECC method, all the test methods showed high precision, having relative standard deviations well below 3%. Compared with the assumed true value (FAS by MC^{max} method), the FAS values determined using GP method are 6.65 % higher due to the inclusion of open pores. TMECC method yielded FAS values dramatically lower than the other methods because of problems with the procedure. As predicated PD method yielded the highest FAS

value because it measured intra-particle voids including open and closed pores. As the PD method includes closed pores, and the GP method excludes these, the difference between the methods represents an estimate of the closed pore voids within the woodchips (4.93 %).

Test duration and cost are two most often concerns for users of a method. As shown in Table 4-6, the quickest method was the GP method (0.5 hrs) and the most time-consuming method was the TMECC method (6-8 hrs). However, the cost for building or purchasing a gas pycnometer is pretty high (approximate \$25,000; Micromeritics Company, 2001) for GP method. Other FAS methods only used common apparatus (e.g. flasks, sieves, heaters, etc) and hence had low cost.

Reliability of methods was determined by their limitation and defects. As discussed (section 4.1.2.1 to 4.1.2.4), all but the PD method had different kinds of limitations or defects. Specifically, TMECC method had problem with its ineffective procedure, GP method had interference from moisture content and MC^{max} method affected by loss of fine particles. For these reasons, PD method was considered as highly reliable while TMECC method produced unacceptable low results. Although having some interference, GP and MC^{max} method was still reliable due to its reasonable results and effective procedures.

As discussed, FAS measured by MC^{max} method was assumed to be the closest to the true value. Also, based on the above comparison, the method had high

precision, low cost, acceptable test duration and good reliability. Although GP method showed its quickness and precision, its main shortcomings are: 1) FAS value included open-pores; 2) moisture interference; and 3) high cost. Therefore, MC^{max} method was selected as the potential method to be further developed in this experiment. Procedure standardization of MC^{max} method would be conducted in Phase II.

4.2 Results and Discussion of Phase II

Phase II was conducted to develop a standard procedure for MC^{max} method that can accurately determine FAS of composting materials. According to the FAS calculation model of MC^{max} method, determination of maximum MC (MC^{max}) was the core part controlling method accuracy. Therefore, in this experiment, standard procedure of MC^{max} method was developed according to the Standard Method 1040: method development and evaluation (APHA 1995).

Standard Method 1040 includes two parts: method validation and collaborative testing, but only method validation was done in this experiment. Method validation includes single-operator characteristics, analysis of unknown sample, method ruggedness and equivalency test. According to the standard method 1040, equivalence test for the MC^{max} was unnecessary because there was no other existing standard method of MC^{max} determination. In the following, detailed analysis of MC^{max} method was conducted on single-operator characteristics, analysis of unknown sample and method ruggedness.

4.2.1 Description of Standardized MC^{max} Method

MC^{max} method was written in the format of Standard Method (APHA 1995), and it consists of general discussion, apparatus, procedure, calculation, and precision. The detailed description of this method was presented in the following:

1. General discussion

- a. *Principle*: Sample is saturated by boiling water to replace all air space in the sample particles. Drain sample and remove excess water attached to the particles, so that the water content inside the particles can be measured by oven-drying. The resulting water content is the maximum that the particles can hold.
- b. *Interference*: small wet particles attached to the sponge cause the loss of sample.

2. Apparatus

- a. *Apparatus for sample water-saturation* (Klute et al. 1986): 1000-mL flask; electric heater or gas burner; analytic balance (weigh to 0.01 g).
- b. *Sieve combination*: 1/32 inch sieve with catch pan and lid
- c. *Sponge*: Kitchen-use handy-size sponge (14 × 9 × 4.5 cm) and cellulose sponge cloth (20.6 × 18.4 × 1.1 cm)
- d. *Paper towel*: Scott® single-fold towels (Kimberly-Clark®)

e. Aluminum plate: for sample collecting after blotting

f. Apparatus for TS determination (standard method 2540 B, APHA 1995): porcelain dish (100-mL); desiccator; 103 °C drying oven; analytical balance (0.01 g).

3. Procedure

a. Sample preparation: apply random sampling methodology to yield about 25 g sample; use 1/32 inch sieve to remove fine particles from the dry sample before water-saturation.

b. Sample water-saturation: refer to the particle density determination procedure (Klute *et al.* 1986)

c. Preparation of sieve combination: put the catch pan on the bottom of 1/32 inch sieve and cover it with lid.

d. Preparation of sponges: use tap water to wet sponge pad and handy size sponge and squeeze them until no water drops out.

e. Sample collecting and draining: transfer the sample from flask to the screen; use tap water to flush the flask several times so that all samples are collected in the screen combination; drain off water from the water-saturated sample for 5 minutes; keep the lid on the screen to avoid water vapour loss.

f. preparation of blotting: place multi-layer paper towels (6-8 pieces) under the sponge pad to prevent it being too wet during blotting operation; transfer

suitable amount of drained sample from the sieve to the sponge pad; make the sample a uniform single thin layer;

g. Blotting: use the pre-wetted sponge to press the sample to adsorb the water outside of sample particles; repeat the operation 3 to 5 times until no water glistening on the sample particle surface; change paper towel and squeeze the sponge once they are wetted.

h. MC^{max} sample preparation: using sponge to brush the sponge pad to remove all blotted samples into a large aluminum pan; cover the aluminum pan to avoid water vapor loss; Repeat the above operation to yield about 25 g samples for MC analysis for each original sample

i. MC^{max} determination: follow the standard method 2540B (APHA, 1995) to determine the total solids (TS) content of the sample; and then calculate moisture content (MC) by TS results.

j. FAS determination: follow the calculation model developed by Eftoda and McCartney (2002) to determine FAS of sample. Data of bulk density, particle density, and moisture content were measured separately in other experiments.

4. Calculation

a. MC^{max} determination:

$$\%TS = \frac{C - B}{A - B} \times 100\%$$

$$\%MC^{max} = 100 - \%TS$$

Where:

A = weight of wet sample + dish, g;

B = weight of dish, g;

C = weight of dry sample + dish, g;

TS = total solids; and

MC^{max} = maximum moisture content.

b. FAS determination:

Refer to FAS calculation model developed by Eftoda and McCartney (2002),
i.e. Equation 3-11, 3-12, 3-13, and 3-14 presented in section 3.1.4 (P.46).

5. Precision

Single-laboratory triplicate analysis of four samples of bulking agents (6.35 – 12.70 mm woodchips; 0 – 19.05 mm woodchips; straw; leaves; and wood shavings) was made with relative standard deviations ranging from 0.11 to 2.02 %.

4.2.2 Method Development and Validation

4.2.2.1 Single-operator Characteristics

This part was to determine the bias and precision of MC^{max} method. According to Standard Method 1040 (APHA 1995), at least 7 but preferably 10 samples are

required to make the determinations of single-operator characteristics. In this experiment, 10 samples of woodchips with size of 6.35 – 12.70 mm were tested using MC^{max} method to determine their MC^{max}. Results are shown in Table 4-7.

From Table 4-7, the average MC^{max} of 10 samples was 65.46 %, which was approximately twice higher than its background MC of 31.56 %. The difference between them indicated that intra-particle voids of woodchips were considerable. RSD of 10 measurements by MC^{max} method was well below 3 %, therefore the method had a good precision. However, it was hard to determine its accuracy because there was no existing true value of MC^{max} for calculating its bias.

4.2.2.2 Unknown Sample Analysis by Second Operator

The purpose of this step in method validation procedure was to apply the developed method to unknown sample and test its deviation from the mean value of standard. APHA (1995) suggested that the mean value of unknown sample recovered should be within 3 standard deviations (*s*) of the mean value of the standard but preferably 2 *s*.

Unknown sample for second operator was 6.35 – 12.70 mm full-scale fine woodchips, and the operator followed procedures of MC^{max} method to perform the test without verbal instructions. Results of this experiment were presented in

Table 4-7. In this experiment, average MC^{max} (65.46 %) of 10 samples (6.35 – 12.70 mm full-scale fine woodchips) measured by first operator was treated as the standard value. According to the suggestion by APHA (1995) mentioned above, the average value of unknown sample in this experiment should be within the range of 63.30 to 67.62 % (i.e. 65.46 % \pm 3 \times 0.72 %) but preferably 64.02 to 66.90 % (i.e. 65.46 % \pm 2 \times 0.72 %).

From Table 4-7, the average MC^{max} measured by second operator was 64.7 %, which was within 2 s of the mean value of standard (64.02 to 66.90 %). The accordance between value of unknown sample and standard revealed that the method developed had good repeatability and suitability. However, it was noticed that standard deviation of MC^{max} by second operator was 1.36 %, which was higher than that (0.72 %) of the 10 samples analysis by first operator. It may be caused by the different amount of replicates (10 for first operator and 3 for second operator) and operation error by different operators. No statistical significant difference was found between results by first and second operators (Appendix D-7; P.151).

Table 4-7 MC^{max} of woodchips (6.35 – 12.70 mm) by different operators

Item	Number of Samples	MC ^{max} (%)	RSD (%)
Single-operator characteristic (by first operator)	10	65.46	1.11
Unknown sample analysis (by second operator)	3	64.70	2.10

4.2.3.3 Method Ruggedness

Method ruggedness was to determine stability of the method under various conditions. A properly conducted ruggedness test points out those procedural steps in which rigor is critical and those in which some leeway is permissible (APHA 1995). In this method, the main procedures included screening, water saturation, draining, blotting and MC determination. Among them, water saturation and MC determination were guided by recognized methods: particle density method (Klute *et al.* 1986) and standard methods: Standard Method 2540B (APHA 1995), respectively. Therefore, the possible variations of procedural steps were screening, draining and blotting. In addition, the type of materials was also considered as a variation factors affecting the MC^{max} determination. All factors with its normal and variation conditions were listed in Table 4-8.

Table 4-8 Factors affecting MC^{max} determination

Factors	Normal	Variation
Screening	Yes ^[1]	No
Draining time	5 minutes	15, 30, and 60 minutes
Blotting	Yes	No
Type of material	Woodchips (<19.05 mm)	Straw and wood shavings

^[1] Screened by 0.79 mm sieve to remove very fine particles that affect blotting

Screening

During the blotting operation, small particles would enter into the openings of sponge so as to affect blotting effects. The purpose of sample screening by 0.79 mm sieve was to remove small particles and thus reduce its influence on MC^{max} determination. In order to investigate the impact from small particles on the MC^{max} determination, screened and unscreened samples (triplicates) were tested and compared in this experiment and the result was summarized in Table 4-9. The average MC^{max} were 64.11 % and 63.61 % for screened and unscreened full-scale fine woodchips, respectively, and the difference between them was only 0.78 %. From statistical report D-8 (P.152), there was no statistically significant difference between MC^{max} of blotted and unblotted samples. Consequently, it can be concluded that the screening was not a critical factor affecting MC^{max} measurement.

Draining Time

After the sample was saturated in the water pycnometer, draining was the first step to remove excess water in the sample/water mixture. Too short a time may not drain excess water and too long a time may cause water loss from inside particles. A suitable draining time needed to be determined for the standard procedure development. For this purpose, varied draining times of 15, 30, and 60 minutes were used for fine WC ($0.79 < \text{size} < 19.05$ mm) to compare with the normal draining time (5 minutes), and the result was presented in Table 4-7. Although the MC^{max} results showed a decline trend with increasing draining time

(5min: 64.11 %, 15 min: 64.60 %, 30 min: 64.39 %, and 60 min: 63.39 %), one way analysis of variance (Appendix D-2; P.146) indicated that there was no statistically significant difference among four groups of results. Therefore, it can be concluded that draining time did not significantly affect the MC^{max} measurement and a 5-minute draining time was good enough.

Blotting

In the MC^{max} method, sponge blotting was adopted to remove excess water between WC particles. To know the importance of blotting operation in MC^{max} method, two groups of WC ($0.79 < \text{size} < 19.05$ mm) samples were blotted with and without sponge in this experiment. Results of these two groups of woodchips were shown in Table 4-7. Both samples used in this test were drained for 5 minutes.

The sample treated with sponge blotting had an average MC^{max} of 64.11 %, while that without blotting treatment had a MC^{max} as high as 70.77 %. The difference of MC^{max} was approximately 10 % between the two groups of WC samples. In addition, the MC^{max} difference caused by blotting was not only reflected by WC samples but also wood shavings samples. As shown in Table B-2-5 (P.125), unblotted wood shavings had a MC^{max} of 83.21 %, while it was only 72.71 % for the blotted one. For both woodchips and wood shavings, t-test (Appendix D-3; P.147) was run for results of blotted and un-blotted samples and it showed statistically significant difference. Moisture difference can also be observed from the physical

phenomenon. Un-blotted samples had water glistening on surface while blotted sample did not have. Overall, it indicated that samples without blotting treatment contained excess water and blotting operation was an important step to remove excess water.

Table 4-9 MC^{max} of woodchips (6.35 – 12.70 mm) determined under different conditions

Conditions	Number of samples	MC ^{max} (%)	RSD (%)
Screened, 5-min draining and blotted	3	64.11	1.55
Unscreened (5-min draining and blotted)	3	63.61	1.57
Un-blotted (screened and 5-min draining)	3	70.77	0.12
15 min draining (screened and blotted)	3	64.60	1.31
30 min draining (screened and blotted)	3	64.39	0.79
60 min draining (screened and blotted)	3	63.39	0.18

Type of Materials

To investigate the applicability of MC^{max} method, different types of materials were used in this experiment. In addition to woodchips, other popular bulking agents, such as straw, wood shavings and leaves, as well as mixture of WC/BS were used. MC^{max} results for these materials were presented in Table 4-10.

For straw, wood shavings and leaves, RSD of MC^{max} were very low and it indicated that MC^{max} method was precise for different bulking agents. However, their MC^{max} were much higher than that of woodchips. It was probably caused by their different internal structure, i.e. amount of micro-pores inside the particle. However, no literature was found to compare the internal structure of different materials. To quantify micro-pores, further research may be conducted to compare FAS value by PD and MC^{max} method.

Unfortunately, MC^{max} method was not performed successfully for woodchips/biosolids mixture because of the loss of considerable wet solids (mostly biosolids). Specifically, the influences from the loss of solids were: 1) changed the original constituent of sample and 2) reduced the water-absorption ability of sponge and hence the blotting effect. Finally, confidence level of MC^{max} measured in this test was affected. For this reason, MC^{max} method needed improving for composting mixtures containing fine particles like biosolids. This work was conducted in Phase III.

Table 4-10 MC^{max} of different types of materials

Materials	Number of Samples	MC^{max} (%)	RSD (%)
Straw	3	83.45	0.12
Wood shavings	3	72.71	0.50
Leaves	3	80.78	0.62
Woodchips/biosolids ^[1]	3	64.75 ^[2]	1.16

^[1] Size of woodchips (6.35-12.70 mm)

^[2] Result may be invalid due to large amounts of solids lost

4.2.4 Summary of Phase II

Phase II completed the MC^{max} method development and applications to different materials. The following summary can be made:

- Established a standard procedure of MC^{max} method according to Standard Method 1040 (APHA, 1995)
- Conducted the method validation and determined that the most important factor affecting the method was blotting.
- Applied MC^{max} method to different materials: woodchips, straw, wood shavings, and woodchips/biosolids mixture. Precise results (RSD % < 3) were obtained for all materials but woodchips/biosolids mixture due to the loss of considerable fine wet solids.
- MC^{max} method needs improving to test composting mixtures containing fine wet solids.

4.3 Results and Discussion of Phase III

Phase III was to improve MC^{max} method developed in Phase II so that it can be applied to compost mixtures. The limitation of MC^{max} method was quantitatively analyzed and a new FAS determination model was developed.

4.3.1 Limitation of MC^{max} Method

MC^{max} method showed satisfactory precision and effectiveness in FAS determination of bulking agents (e.g. woodchips, wood shavings, straw, leaves, etc.), but was not suitable for composting mixtures because of the interference from fine particles. Although the loss of considerable particles was observed in Phase II experiment, the quantitative analysis was not performed. To conduct the investigation, MC^{max} method was applied to woodchips/mixture (size of woodchips < 19.05 mm) with MC of 44.4 %, and the results were presented in Table C-1 (P.135).

In this experiment, it was observed that fine solids lost were from both biosolids and woodchips. Solids lost by draining were mostly biosolids while those lost by blotting include both biosolids and fine particles of woodchips. Although solids lost were also observed for woodchips samples (in Phase II), it was much worth

for woodchips/mixtures because biosolids attached on sponge easily clung fine woodchips.

The analytical results in Table C-1 (P.135) showed that total dry solid loss was 31.5 % (dry basis) and the loss by blotting was 22.2 % (dry basis). The considerable solid loss may change the component of the sample and thus cause low confident level results. From Table C-1, MC^{max} of woodchips/biosolids mixture determined by MC^{max} method was 65.4 %, which was just a little bit greater than that of woodchips (63.61 %). In the water-saturated mixture, biosolids and woodchips should have the same MC (65.40 %). Compared with its original MC (71.95 %), MC^{max} of biosolids was lower and was deemed unacceptable.

The large amount of solid loss during the process and abnormal MC^{max} indicated that the MC^{max} method was ineffective to deal with composting materials containing sticky fine particles. Without question, the method would also not be suitable for other compost mixtures, such as straw/biosolids or leaves/biosolids. For this reason, MC^{max} method needed improving to suit all composting materials.

4.3.2 Development of New FAS Determination Model

As discussed on the above, two key procedures: draining and blotting, contributed to the solid loss and MC^{max} deviation of woodchips/biosolids. Short-time draining

and blotting, however, were necessary to remove excess water of sample. Obviously, these two procedures can not be changed. Since the procedure could not be changed to solve the problem, a new FAS numerical model was required.

Fortunately, a conceptual model (Haug, 1993) for estimating volumetric mixing ratio of composting mixtures was found useful to determine FAS of composting mixtures. This model was established on the theory of moisture transfer between wet substrate and bulking agents. Six assumptions were made for this model and they were met by composting mixtures used in this experiment:

- 1) Wet substrate is semi-fluid and occupies the interstices of the mixture

Conditions met: MC of biosolids was high as 71.95 % and it occupies interstices of mixture

- 2) Sufficient bulking agents to keep mixture porous

Conditions met: Air-filled porosity or TAS of mixtures ranged from 51.33 to 70.97% (refer to Table C-5-1, C-5-2, and C-5-3; P.140)

- 3) Moisture absorption by bulking agents was limited to a maximum MC

Conditions met: Maximum MC of woodchips (0-19.05 mm): 63.61 %; woodchips (1/4 – 1/2): 65.46 %; Straw: 83.45 %

- 4) Moisture release from wet substrate is limited to a maximum solid content

Conditions met: Moisture release from wet substrate is limited to 45% (solid content) due to target MC of mixture is 55 %

- 5) Amount of other amendments added have to be small enough to not violate the first assumption

Conditions met: No other amendments added

- 6) Individual bulking agents particles are solid with no internal FAS (i.e. internal FAS was not considered as part of FAS of bulking agents because of its little value to oxygen transfer)

Conditions met: FAS defined in this paper does not include internal air space

Based on MC^{max} method and the rearranged Haug's conceptual model (Equation 3-16), a FAS numerical model was established for composting mixtures. In order to test its effectiveness and suitability, the FAS numerical model was used for two kinds of popular composting mixtures: woodchips/biosolids and straw/biosolids. FAS determined by the numerical model were listed in Table 4-11. Also, FAS measure by PD method was cited in bracket for comparison purpose.

Table 4-11 FAS of composting mixtures by the FAS numerical model

Materials	Size of bulking agents (mm)	Number of samples	FAS (%)	RSD (%)
WC/BS	0 - 19.05	2	39.62 (51.33*)	0.71
	6.35 - 12.7	2	48.55 (61.76*)	0.46
Straw/BS	Not determined	2	31.53 (70.93*)	3.08

* FAS measured by PD method

As shown in Table 4-11, FAS determined by FAS numerical model were much lower than those by the traditional method (PD method). Statistical analysis (Appendix D-12; P.156) showed that there were statistically differences for FAS determined by new model and PD method for all mixtures. Specifically, FAS for WC/BS and straw/BS mixtures by the FAS numerical model were 12 % and 40 %, respectively, which were much lower than those by PD method.

The differences between the FAS determined by the two methods can be explained by their different FAS definitions. As mentioned, FAS measured by PD method included inter- and intra-particle voids, which actually is total air space (TAS). The new determination model was developed for only inter-particle voids determination, which is the FAS defined in this paper. Therefore, the main difference between FAS by new determination model and PD method was the intra-particle voids. Referring to Table B-5-1 and B-5-2 (P.131), the volumetric ratio of air (i.e. intra-particle voids) to sample for two kinds of woodchips were 20 % (size: 6.35 – 12.70 mm) and 24 % (size: 0- 19.05 mm), respectively. Therefore, FAS differences by new model and PD method for woodchips/biosolids mixtures (6.35 -12.70 mm: 13.21 % and 0-19.05 mm: 11.71 %) were a part of intra-particle voids.

For straw/BS mixture, FAS difference by new determination model and PD method was high as 39.4 %. This difference can not be simply explained by the volumetric ratio of intra-particle voids to sample, because straw only had 27 % of

volumetric ratio of air (inside micro-pores) to sample (referred to Table B-5-3; P.131). Another possible reason for the difference may be the property and amount of straw. Compared with woodchips, straw was very soft and easy to be squeezed together. Moreover, it was confirmed by sharply reduced volume after mixing. From Table C-6-3 (P.143), volume of straw was 9.9 L and volume of straw/biosolids was only 5.7 L. In addition, if the amount of straw was insufficient, it would immerse in biosolids after mixing so that it could not provided enough porous structure for the mixture (Haug, 1993). Therefore, low FAS of straw/biosolids may relate to the high ratio of intra-particle voids, strength, and insufficient amount of bulking agents.

Although the new FAS determination model showed its ability and precision (low RSD) to determine FAS of composting mixture, further work were required to determine its accuracy and suitability for a broad range of materials.

Summary, Conclusions and Recommendations

The purpose of this research was to develop a standard method for FAS determination that suits all composting materials. The research was started from the clarification of FAS definition and comparison of current available methods, and then focused on the procedure standardization for the selected method: MC^{max} method. The procedure of MC^{max} method was developed and standardized according to the standard method 1040: method development and evaluation (APHA 1995). The developed method was applied to different bulking agents and obtained precise FAS results, but it was found ineffective for determining FAS of composting mixtures containing fine particles. In order to overcome the shortage, a FAS numerical model that combines MC^{max} method and Haug's conceptual model (Haug, 1993) was proposed and applied to some composting mixtures. The FAS determination model showed its ability to determine FAS of composting mixture but further work were required to determine its accuracy and suitability for *in-situ* composting environment.

Based on the results and discussions, the following conclusions can be drawn:

1. Total air space (TAS) in a compost matrix include inter- and intra- particle voids, and free air space (FAS) is defined as inter-particle voids. As an important parameter in composting process, FAS of compost is limited by

many physical conditions: moisture content, particle size, bulk density, types of bulking agents, as well as the location in a pile.

2. Current available methods for measuring FAS, e.g. the particle density (PD) method, test methods for the examination of composting and compost (TMECC): 03.01 A & B (USCC, 2001), gas pycnometer (GP) method and maximum moisture content (MC^{max}) method, were analyzed in Phase I for their effectiveness and suitability. Except TMECC method, all other three methods showed their high precision ($RSD < 3\%$) in measuring FAS. Defects or limitations were found for all methods: 1) FAS measurements include intra-particle voids (for both PD and GP method); 2) method defects: insufficient water-saturation and blocking of draining hole (for TMECC method 03.01 A & B); 3) Chamber size and MC interference (for GP method); and 4) Loss of fine particles (for MC^{max} method).
3. Based on the results of the same material (3.18-9.53 mm woodchips), all FAS measuring methods were compared for precision, bias, test duration, cost, and reliability. Although PD method showed its reliability and precision, its FAS value included intra-particle voids. TMECC method was considered as ineffective due to the problem with its procedure. GP method was quick and precise but its measurement includes open-pores and it required high-cost equipment. For a comprehensive consideration, MC^{max} method was selected for further development due to its FAS value was the mostly close to the true

value and it had high precision, low cost, acceptable test duration and good reliability.

4. Factors affecting MC^{max} method include screening, draining time, and blotting.

It was proved by statistical analysis that blotting is the key factors for MC^{max} method. For woodchips, un-blotted sample has 10 % higher MC^{max} than blotted one.

5. MC^{max} method showed its high precision (% RSD for triplicate samples < 2) in FAS determinations for different kinds of bulking agents. However, it was not able to deal with materials containing fine wet particles. The application of MC^{max} method to woodchips/biosolids mixture showed that the percentage of dry solid lost during the process was as high as 32 % and MC^{max} (65.24 %) of mixture was lower than initial MC (71.95 %) of biosolids.

6. To solve the shortcomings of the MC^{max} method, a FAS numerical model was developed by combining the MC^{max} method and Haug's conceptual model. The FAS numerical model showed its ability to determine FAS of composting mixture containing fine particles. Significant differences existed between FAS by the FAS numerical model and the traditional PD method. For two kinds of woodchips/biosolids, the differences (approximate 12 %) were mainly caused by micro-pores (intra-particle voids). For straw/biosolids, strength and

amount of straw were also considered as the possible reasons for its low FAS (39 % lower).

7. The engineering significance of using a standard FAS method includes: 1) designing the recipe for a composting pile with the desired FAS level; 2) maintaining sufficient oxygen supply and heat retention; and 3) reducing the potential of anaerobic condition and odor.

Although the FAS numerical model showed its ability to determine FAS of composting mixtures, further studies are required to apply it to a broad range of composting materials and investigate factors affecting its accuracy. Field research needs to be conducted to determine its suitability and effectiveness for *in-situ* composting environment. Furthermore, the new FAS determination model had the potential for field project to estimate the ratio of substrate to bulking agents required to reach any desired FAS in a composting mixture. In addition, GP method would be an accurate and powerful method to measure FAS if the deviation caused by MC and open pores were figured out.

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Appendix A

Phase I Raw Data

Table A-1-1 Moisture content (MC) of full-scale fine woodchips (0 - 19.05 mm)

Sample	Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Weight of dish after heating at 105 °C (g)	Weight of sample after heating at 105°C (g)	TS (%)	Moisture (%)	Average moisture (%)	STDEV (%)
1	88.35	103.81	15.46	99.02	10.67	69.02	30.98	31.51	1.32
2	90.96	109.53	18.57	103.40	12.44	66.99	33.01		
3	87.91	108.48	20.57	102.20	14.29	69.47	30.53		

Table A-1-2 Moisture content (MC) of sieved full-scale fine woodchips (3.18 - 9.53 mm)

Sample	Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Weight of dish after heating at 105 °C (g)	Weight of sample after heating at 105°C (g)	TS (%)	Moisture (%)	Average moisture (%)	STDEV (%)
1	85.18	102.37	17.19	95.72	10.54	61.31	38.69	38.47	0.19
2	89.90	106.21	16.31	99.96	10.06	61.68	38.32		
3	96.67	110.41	13.74	105.11	8.44	61.43	38.57		
4	14.98	231.56	216.58	148.64	133.66	61.71	38.29		

Table A-1-3 Moisture content (MC) of Biosolids

Sample	Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Weight of dish after heating at 105 °C (g)	Weight of sample after heating at 105°C (g)	TS (%)	Moisture (%)	Average moisture (%)	STDEV (%)
1	22.45	140.4	117.95	55.74	33.29	28.22	71.78	71.95	0.24
2	22.37	150.58	128.21	57.98	35.61	27.77	72.23		
3	23.21	159.33	136.12	61.53	38.32	28.15	71.85		

Table A-1-4 Moisture content (MC) of composting mixtures

Sample	Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Weight of dish after heating at 105 °C (g)	Weight of sample after heating at 105°C (g)	TS (%)	Moisture (%)	Average moisture (%)	STDEV (%)
WC/BS-1	25.44	36.33	10.89	31.10	5.66	51.96	48.04	49.48	1.68
WC/BS-2	24.42	32.96	8.54	28.76	4.35	50.91	49.09		
WC/BS-3	27.30	36.61	9.32	31.83	4.53	48.68	51.32		
LV/BS-1	28.26	39.97	11.71	33.92	5.65	48.28	51.72	51.30	0.45
LV/BS-2	25.61	34.36	8.75	29.91	4.30	49.17	50.83		
LV/BS-3	26.88	35.54	8.65	31.09	4.21	48.64	51.36		

Note: 1) WC/BS: woodchips/biosolids mixture and the size of woodchips (3.18 - 9.53 mm)

2) LV/BS: leaves/biosolids mixture

Table A-3-1 Bulk density (BD) of full-scale fine woodchips (0 - 19.05 mm)

Compressive Cell	Number	Weight of Cell (g)	Weight of Cell and Sample (g)	Weight of Sample (g)	Volume of Cell (cm ³)	Wet BD (g/cm ³)	Average Wet BD (g/cm ³)	Moisture Content (%)	Average Dry BD (g/cm ³)	STDEV
Small	S1	1640	2510	870	3532.50	0.2463	0.2365	31.5100	0.1620	0.0171
	S2	1650	2550	900	3532.50	0.2548				
Medium	M1	9940	14210	4270	19625.00	0.2176				
	M2	8930	13390	4460	19625.00	0.2273				

Table A-3-2 Bulk density (BD) of sieved full-scale fine woodchips (3.18 -9.53 mm)

Sample (dry)	Weight of Cup (g)	Weight of CUP & WC (g)	Weight of WC (g)	Volume of Cup (cm ³)	Dry BD (g/cm ³)	Average Dry BD (g/cm ³)	Average moisture (%)	Wet bulk density (g/cm ³)	Average wet bulk density (g/cm ³)	STDEV
1	41.816	55.371	13.555	142.5	0.095	0.09774	38.47	0.1546	0.1588	0.0060
2	41.816	56.117	14.301	142.5	0.100			0.1631		

Table A-3-3 Bulk density (BD) of woodchips (Gagne 2001)

Biocell	Weight of Cell (g)	Weight of Cell and Sample (g)	Weight of Sample (g)	Wet BD (g/cm ³)	Average Wet BD (g/cm ³)	Moisture Content (%)	Dry BD (g/cm ³)	Average Dry BD (g/cm ³)	STDEV
1	1639.9	2450.6	810.7	0.223	0.22945	41.0300	0.13173	0.13531	0.003439
2	1637.2	2475.4	838.2	0.231			0.136198		
3	1630.2	2489.8	859.6	0.237			0.139676		
4	1656.7	2479.1	822.4	0.227			0.133631		

Table A-3-4 Bulk density (BD) of biosolids/woodchips (size of woodchips: 3.18 - 9.53 mm)

Biocell	Weight of Cell (g)	Weight of Cell and Sample (g)	Weight of Sample (g)	Volume of Cell (cm ³)	Wet BD (g/cm ³)	Average Wet BD (g/cm ³)	Moisture Content (%)	Average Dry BD (g/cm ³)	STDEV
1	1658.5	2957.6	1299.1	3532.50	0.3678	0.3814	49.48	0.1927	0.0193
2	1655.6	3051.2	1395.6	3532.50	0.3951				

Table A-4-1 Particle Size Analysis of Full-scale Coarse Woodchips

Sieve Size		Tare (g)	Sample # 1				Sample # 2				Average Σ %
			Tare + Coarse (g)	Coarse (g)	%	Σ %	Tare + Coarse (g)	Coarse (g)	%	Σ %	
1/16	1.59	285.1	305.5	20.4	6	6	298.8	13.7	6	6	6
1/8	3.18	472.3	499.2	26.9	9	15	491.9	19.6	8	14	14
1/4	6.35	607.9	627.8	19.9	6	21	621.6	13.7	6	19	20
3/8	9.53	541.9	575.7	33.8	11	32	573.8	31.9	13	33	32
1/2	12.70	558.5	606.8	48.3	15	47	584.6	26.1	11	43	45
3/4	19.05	553.2	609	55.8	18	65	590.1	36.9	15	59	62
1	25.40	573.3	600.9	27.6	9	74	603.0	29.7	12	71	72
1 1/4	31.75	657.9	662	4.1	1	75	673.8	15.9	7	77	76
1 1/2	38.10	585.5	594.3	8.8	3	78	609.7	24.2	10	87	83
2	50.80	528.2	598.1	69.9	22	100	558.5	30.3	13	100	100
Total				315.5	100			242	100		

Table A-4-2 Particle Size Analysis of Full-scale Fine Woodchips

Sieve Size		Tare (g)	Sample # 1				Sample # 2				Average Σ %
			Tare + Coarse (g)	Coarse (g)	%	Σ %	Tare + Coarse (g)	Coarse (g)	%	Σ %	
1/32	0.79	285.1	309.2	24.1	8	8	310.4	25.3	8	8	8
1/16	1.59	450.3	480.7	30.4	10	18	480.9	30.6	10	18	18
1/8	3.18	472.3	581.3	109	36	54	574.5	102.2	33	50	52
1/4	6.35	607.9	655.5	47.6	16	70	660.7	52.8	17	67	69
3/8	9.53	541.9	599.2	57.3	19	89	609.1	67.2	21	89	89
1/2	12.70	558.5	577.7	19.2	6	96	580.3	21.8	7	96	96
3/4	19.05	553.2	566.7	13.5	4	100	567.1	13.9	4	100	100
1	25.40	573.3	573.3	0	0	100	573.3	0	0	100	100
1 1/4	31.75	657.9	657.9	0	0	100	657.9	0	0	100	100
1 1/2	38.10	585.5	585.5	0	0	100	585.5	0	0	100	100
2	50.80	528.2	528.2	0	0	100	528.2	0	0	100	100
Total				301.1	100	Total		313.8	100		

Table A-5-1 Particle density of woodchips (0 - 19.05 mm)

Sample	Weight of empty flask W_p (g)	Weight of flask and sample (g)	Sample moisture content (%)	Weight of flask and sample corrected to oven dried weight W_s (g)	Weight of flask and sample, filled with boiled water W_{sw} (g)	Weight of flask and boiled water W_w (g)	Sample particle density PD (g/cm ³)	STDEV
1	234.5	276.2	30.98	263.28	1229.8	1218.2	1.67	0.05
2	244.3	284.6	30.98	272.12	1244.4	1232.9	1.70	
3	246.3	286.5	30.98	274.05	1242.9	1232.4	1.60	
Temperature at which measurements were taken =				22.5	°C			
Density of water at which measurements were taken =				0.9976	g/cm ³			
$PD = p_w(W_s - W_p) / [(W_s - W_p) - (W_{sw} - W_w)]$								
Average PD =		1.66	g/cm ³					

Table A-5-2 Particle density of sieved full-scale fine woodchips (3.18 - 9.53 mm)

Sample	Weight of empty flask W_p (g)	Weight of flask and sample (g)	Sample moisture content (%)	Weight of flask and sample corrected to oven dried weight W_s (g)	Weight of flask and sample, filled with boiled water W_{sw} (g)	Weight of flask and boiled water W_w (g)	Sample particle density PD (g/cm ³)	STDEV
1	243.6	273.4	0	273.40	1238.5	1229	1.46	0.11
2	242.7	275.6	0	275.60	1244.8	1231.5	1.67	
3	238.1	273.4	0	273.40	1234.5	1222.7	1.50	
Temperature at which measurements were taken =				28.9	°C			
Density of water at which measurements were taken =				0.9959	g/cm ³			
$PD = p_w(W_s - W_p) / [(W_s - W_p) - (W_{sw} - W_w)]$								
Average PD =		1.54	g/cm ³					

Table A-5-3 Particle density of biosolids/woodchips (Size of woodchips: 3.18 - 9.53 mm)

Sample	Weight of empty flask W_p (g)	Weight of flask and sample (g)	Sample moisture content (%)	Weight of flask and sample corrected to oven dried weight W_s (g)	Weight of flask and sample, filled with boiled water W_{sw} (g)	Weight of flask and boiled water W_w (g)	Sample particle density PD (g/cm ³)	STDEV
1	234.6	265.1	0	265.10	1232.1	1218.2	1.83	0.02
2	244.3	274.9	0	274.90	1247.2	1232.9	1.87	
3	246.3	276.5	0	276.50	1246.3	1232.4	1.85	
Temperature at which measurements were taken =				22.5	°C			
Density of water at which measurements were taken =				0.99757	g/cm ³			
PD = $p_w(W_s - W_p) / [(W_s - W_p) - (W_{sw} - W_w)]$								
Average PD =		1.85	g/cm ³					

Table A-6-1 FAS of full-scale fine woodchips (0 - 19.05 mm) by particle density method

Sample	Average wet bulk density (g/cm ³)	Average moisture (%)	Average dry bulk density (g/cm ³)	Particle density (g/cm ³)	Porosity (%)	Average porosity (%)	Average water content (%)	FAS (%)	Average FAS (%)	STDEV (%)	RSD (%)
1	0.2365	31.51	0.1620	1.67	90.31	90.23	7.45	82.85	82.78	0.29	0.35
2				1.70	90.48			83.02			
3				1.60	89.91			82.46			

Table A-6-2 FAS of sieved full-scale fine woodchips (3.18 - 9.53 mm) by particle density method

Sample	Average wet bulk density (g/cm ³)	Average moisture (%)	Average dry bulk density (g/cm ³)	Particle density (g/cm ³)	Porosity (%)	Average porosity (%)	Average water content (%)	FAS (%)	Average FAS (%)	STDEV (%)	RSD (%)
1	0.1588	38.47	0.0977	1.67	94.15	94.11	6.11	88.04	88.00	0.18	0.20
2				1.70	94.25			88.15			
3				1.60	93.91			87.80			

Table A-6-3 FAS of full-scale coarse woodchips by particle density method

Sample	Average wet bulk density (g/cm ³)	Average moisture (%)	Average dry bulk density (g/cm ³)	Particle density (g/cm ³)	Porosity (%)	Average porosity (%)	Average water content (%)	FAS (%)	Average FAS (%)	STDEV (%)	RSD (%)
1	0.1583	21.36	0.1245	1.62	92.32	92.81	3.38	88.93	89.43	0.70	0.78
2				1.86	93.31			89.93			

Note: Wet BD, average MC and PD refer to Eftoda (2002)

Table A-6-4 FAS of lab-scale fine woodchips by particle density method

Sample	Average wet bulk density (g/cm ³)	Average moisture (%)	Average dry bulk density (g/cm ³)	Particle density (g/cm ³)	Porosity (%)	Average porosity (%)	Average water content (%)	FAS (%)	Average FAS (%)	STDEV (%)	RSD (%)
1	0.2290	41.03	0.1350	1.90	92.89	92.30	9.40	83.49	82.91	1.46	1.76
2				2.24	93.96			84.57			
3				1.43	90.56			81.16			
4				1.65	91.82			82.42			

Note: Wet BD, average MC and PD refer to Gagne (2001)

Table A-6-5 FAS of biosolids/woodchips (sieved full-scale fine woodchips: 3.18 - 9.53 mm)

Sample	Average wet bulk density (g/cm ³)	Average moisture (%)	Average dry bulk density (g/cm ³)	Particle density (g/cm ³)	Porosity (%)	Average porosity (%)	Average water content (%)	FAS (%)	Average FAS (%)	STDEV (%)	RSD (%)
1	0.3814	49.48	0.1927	1.83	89.49	89.59	18.87	70.62	70.72	0.11	0.16
2				1.87	89.71			70.84			
3				1.85	89.57			70.70			

Table A-7-1 Data record of sample before and after draining in TMECC method

Beaker	WC-1	WC-2	WC-3	LV-1	LV-2	WC/BS-1	WC/BS-2	LV/BS-1	LV/BS-2
Solids content (fractional)	0.41	0.41	0.41	0.41	0.41	0.5	0.5	0.51	0.51
Wt. of empty beaker (g)	141	140.9	141	140.9	141	141.4	141.3	141.4	141.4
Wt. of beaker + sample filled to 1800ml (g)	586.1	540.3	560.8	399.7	375.6	606.2	606.2	632.3	647.9
Volume of water added initially (mL)	1580	1440	1490	1425	1400	1080	1120	1120	1150
Volume of water remaining in catch beaker after final filling (mL)	150	200	170	150	150	500	500	250	300
Wt. of beaker + sample + water after final filling (g)	1699.6	1580	1605.6	1681.3	1615.6	1110.7	1156.9	1512.2	1560.6
Wt. of beaker + sample after final drain (g)	887.6	818.2	845.7	829.2	768.7	854.9	940	1054.9	1078
Water saturated volume after final drain (ml)	1700	1600	1675	1450	1300	1050	1025	1330	
Volume of water remaining in catch beaker after final drain (mL)	900	900	910	1000	950	-	-	-	-

Note: Woodchips were sieved full-scale fine woodchips

Table A-7-2 Calculation of FAS measured by TMECC method

Parameters		WC-1	WC-2	WC-3	LV-1	LV-2	WC/BS-1	WC/BS-2	LV/BS-1	LV/BS-2
V_{1800, after drain}	(mL)	1800.00	1800.00	1801.00	1800.00	1800.00	1800.00	1800.00	1800.00	1800.00
ARW₁₈₀₀	(g)	445.10	399.40	419.80	258.80	234.60	464.80	464.90		506.50
W_{1800, no drain}	(g)	1558.60	1439.10	1464.60	1540.40	1474.60	969.30	1015.60	1370.80	1419.20
W_{1800, after drain}	(g)	746.60	677.30	704.70	688.30	627.70	713.50	798.70	913.50	936.60
ODW₁₈₀₀	(g)	182.49	163.75	172.12	106.11	96.19	232.40	232.45	0.00	258.32
M₁₈₀₀	(g)	262.61	235.65	247.68	152.69	138.41	232.40	232.45	0.00	248.19
PSV₁₈₀₀	(mL)	1376.11	1275.35	1292.48	1434.29	1378.41	736.90	783.15	1370.80	1160.89
PS%	(%)	76.45	70.85	71.76	79.68	76.58	40.94	43.51	76.16	64.49
Average PS %	(%)	73.02			78.13		42.22		70.32	
RSD	(%)	4.11			2.81		4.30		11.73	
FASV_{me}	(mL)	812.00	761.80	759.90	852.10	846.90	255.80	216.90	457.30	482.60
FAS%	(%)	45.11	42.32	42.19	47.34	47.05	14.21	12.05	25.41	26.81
Average FAS %	(%)	43.21			47.19		13.13		26.11	
RSD	(%)	3.82			0.43		11.64		3.81	

Note:

ARW₁₈₀₀: initial weight of 1800 mL sample as-received

ODW₁₈₀₀: oven dried weight of 1800 mL sample

M₁₈₀₀: moisture content of 1800 mL sample

W_{1800, no drain}: weight of saturated sample before 4-hr draining

PS: pore space LV: Leaves FAS: free air space

PSV₁₈₀₀: pore space volume of 1800 mL sample

FAS_{me}: free air space using mass equivalents, mL ~ g

WHC: water holding capacity

W_{1800, after drain}: weight of saturated sample after 4-hr draining

Table A-8-1 FAS of sieved full-scale fine woodchips (3.18 - 9.53 mm) by gas pycnometer method

Sample	Test#	P1 (psi)	P2 (psi)	Volume of sample (cm ³)	Skeletal volume of sample (cm ³)	FAS (%)	Average FAS (%)	STDEV (%)	RSD (%)
Oven-dried (MC = 0 %)	1	19.51	12.87	142.50	10.12	92.90	92.78	0.10	0.11
	2	19.58	12.91	142.50	10.32	92.76			
	3	19.58	12.91	142.50	10.41	92.70			
As-received (MC = 38.47 %)	1	19.41	12.33	142.5	23.71	83.36	83.07	0.36	0.43
	2	19.54	12.375	142.5	24.69	82.67			
	3	19.64	12.466	142.5	23.97	83.18			

Table A-9-1 FAS of sieved full-scale fine woodchips (3.18 - 9.53 mm) by MC^{max} method

Sample	Average wet bulk density (g/cm ³)	Average TAS (%)	Average moisture (%)	Maximum moisture content (%)	Average Maximum Moisture Content (%)	Water content (%)	Volume of air in micro-pores (%)	FAS (%)	Average FAS (%)	STDEV (%)	RSD (%)
1	0.1588	88.00	38.47	64.65	64.42	17.87	11.76	76.24	76.42	0.39	0.51
2				63.78		17.21	11.10	76.90			
3				64.28		17.58	11.47	76.53			
4				64.95		18.10	12.00	76.00			

Note: Sample was blotted using paper towel

Table A-9-2 FAS of lab-scale fine woodchips (blotted and un-blotted using paper towel) by MC^{max} method

Sample	Average wet bulk density (g/cm ³)	Average TAS (%)	Average moisture (%)	Maximum moisture content (%)	Average Maximum Moisture Content (%)	Water content (%)	Volume of air in micro-pores (%)	FAS (%)	Average FAS (%)	STDEV (%)	RSD (%)
Unblotted-1	0.2295	82.91	41.03	70.20	70.04	31.88	22.46	60.45	60.68	0.24	0.39
Unblotted-2				69.89		31.41	21.99	60.92			
Unblotted-3				70.05		31.65	22.24	60.67			
Blotted -1	0.2295	82.91	41.03	67.21	67.43	27.74	18.32	64.59	64.30	0.28	0.43
Blotted -2				67.45		28.05	18.63	64.28			
Blotted -3				67.64		28.29	18.87	64.04			

Note: Sample was blotted using paper towel

Table A-9-3 FAS of leaves by MC^{max} method

Sample	Average wet bulk density (g/cm ³)	Average TAS (%)	Average moisture (%)	Maximum moisture content (%)	Average Maximum Moisture Content (%)	Water content (%)	Volume of air in micro-pores (%)	FAS (%)	Average FAS (%)	STDEV (%)	RSD (%)
1	0.1532	89.10	38.68	78.51	78.28	34.32	28.39	60.71	61.17	0.40	0.66
2				78.18		33.66	27.73	61.37			
3				78.14		33.59	27.66	61.44			

Note: [1] Wet BD, TAS and average MC of leaves were cited from Gagne (2001)

[2] Sample was blotted using paper towel

Appendix B

Phase II Raw Data

Table B-1-1 Moisture content (MC) of woodchips with different sizes

Sample	Size (mm)	Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Weight of dish after heating at 105 °C (g)	Weight of sample after heating at 105°C (g)	TS (%)	MC (%)	Average MC (%)	STDEV
Large-01	6.35 - 12.70	70.05	88.96	18.91	82.87	12.82	67.79	32.21	31.56	0.58
Large-02		82.38	109.86	27.48	101.24	18.86	68.63	31.37		
Large-03		86.92	116.63	29.71	107.39	20.47	68.90	31.10		
Screened-01	0.79 - 19.05	100.53	130.2	29.67	121.87	21.34	71.92	28.08	29.31	9.77
Screened-02		70.05	88.99	18.94	83.52	13.47	71.12	28.88		
Screened-03		68.67	87.08	18.41	81.38	12.71	69.04	30.96		
unscreened-01	0 - 19.05	82.41	109.08	26.67	101.79	19.38	72.67	27.33	27.99	9.33
unscreened-02		81.04	107.15	26.11	99.46	18.42	70.55	29.45		
unscreened-03		85.12	115.21	30.09	107.03	21.91	72.81	27.19		

Table B-1-2 Moisture content (MC) of straw and wood shavings

Sample	Size (mm)	Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Weight of dish after heating at 105 °C (g)	Weight of sample after heating at 105°C (g)	TS (%)	MC (%)	Average MC (%)	STDEV
Straw-01	unknown	4.02	14.94	10.92	11.85	7.83	71.70	28.30	32.50	4.85
Straw-02		3.97	16.88	12.91	12	8.03	62.20	37.80		
Straw-03		4	16.36	12.36	12.48	8.48	68.61	31.39		
Woodshaving-01	unknown	87.61	99.44	11.83	98.24	10.63	89.86	10.14	10.28	3.43
Woodshaving-02		100.53	113.48	12.95	112.16	11.63	89.81	10.19		
Woodshaving-03		87.52	99.41	11.89	98.16	10.64	89.49	10.51		

Table B-2-1 Maximum moisture content (MC^{max}) woodchips ($6.35 < \text{size} < 12.70 \text{ mm}$)

Sample	Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Weight of dish after heating at 105°C (g)	Weight of sample after heating at 105°C (g)	TS (%)	MC^{max} (%)	Average MC^{max} (%)	STDEV	RSD (%)
1	82.41	130.21	47.8	98.51	16.1	33.68	66.32	65.46	0.72	1.11
2	78.34	126.8	48.46	95.15	16.81	34.69	65.31			
3	81.04	131.37	50.33	98.9	17.86	35.49	64.51			
4	85.12	134.3	49.18	101.59	16.47	33.49	66.51			
5	100.53	150.93	50.4	117.66	17.13	33.99	66.01			
6	86.92	126.57	39.65	100.48	13.56	34.20	65.80			
7	87.06	128.43	41.37	101.58	14.52	35.10	64.90			
8	87.02	131.44	44.42	102.29	15.27	34.38	65.62			
9	82.38	124.49	42.11	97.04	14.66	34.81	65.19			
10	68.69	110.57	41.88	83.6	14.91	35.60	64.40			

Table B-2-2 Maximum moisture content (MC^{max}) of woodchips ($6.35 < \text{size} < 12.70 \text{ mm}$) for second analysis

Sample	Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Weight of dish after heating at 105°C (g)	Weight of sample after heating at 105°C (g)	TS (%)	MC^{max} (%)	Average MC^{max} (%)	STDEV	RSD (%)
Blotted-01	83.76	139.68	55.92	103.44	19.68	35.19	64.81	64.70	1.36	2.10
Blotted-02	89.98	139.03	49.05	106.66	16.68	34.01	65.99			
Blotted-03	85.18	123.75	38.57	99.34	14.16	36.71	63.29			

Table B-2-3 Maximum moisture content (MC^{max}) of full-scale fine WC ($0 < \text{size} < 19.05 \text{ mm}$) under different test conditions

Sample		Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Weight of dish after heating at 105 °C (g)	Weight of sample after heating at 105°C (g)	MC^{max} (%)	Average MC^{max} (%)	Average moisture (%)	STDEV	RSD (%)
Conditions	Number										
Screened (Blotted and 5-min draining)	1	87.05	141.98	54.93	106.72	19.67	35.81	64.19	64.11	1.00	1.55
	2	78.34	124.19	45.85	94.36	16.02	34.94	65.06			
	3	87.24	130.3	43.06	103.14	15.9	36.93	63.07			
Unscreened (Blotted and 5-min draining)	1	82.38	123.77	41.39	97.02	14.64	35.37	64.63	63.61	1.00	1.57
	2	86.92	142.04	55.12	107.52	20.6	37.37	62.63			
	3	81.22	121.03	39.81	95.72	14.5	36.42	63.58			
Not Blotted (Screened and 5-min draining)	1	82.42	160	77.58	105.13	22.71	29.27	70.73	70.77	0.08	0.12
	2	85.13	171.56	86.43	110.44	25.31	29.28	70.72			
	3	100.54	170.43	69.89	120.9	20.36	29.13	70.87			
15-min Draining (Screened and blotted)	1	88.21	139.56	51.35	105.89	17.68	34.43	65.57	64.60	0.85	1.31
	2	82.13	140.56	58.43	103.15	21.02	35.97	64.03			
	3	87.38	145.65	58.27	108.24	20.86	35.80	64.20			
30-min Draining (Screened and blotted)	1	92.18	168.02	75.84	118.86	26.68	35.18	64.82	64.39	0.51	0.79
	2	100.05	178.95	78.9	128.59	28.54	36.17	63.83			
	3	85.91	151.23	65.32	109.09	23.18	35.49	64.51			
60-min Draining (Screened and blotted)	1	78.28	157.35	79.07	107.22	28.94	36.60	63.40	63.39	0.11	0.18
	2	105.33	174.58	69.25	130.76	25.43	36.72	63.28			
	3	99.63	168.26	68.63	124.68	25.05	36.50	63.50			

Table B-2-4 Maximum moisture content (MC^{max}) of straw

Sample	Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Weight of dish after heating at 105 °C (g)	Weight of sample after heating at 105°C (g)	TS (%)	MC ^{max} (%)	Average MC ^{max} (%)	STDEV	RSD (%)
Straw-01	11.29	73.19	61.9	21.6	10.31	16.66	83.34	83.45	0.10	0.12
Straw-02	11.35	77.08	65.73	22.21	10.86	16.52	83.48			
Straw-03	11.35	69.28	57.93	20.89	9.54	16.47	83.53			

Table B-2-5 Maximum moisture content (MC^{max}) of wood shavings (blotted and un-blotted)

Sample	Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Weight of dish after heating at 105 °C (g)	Weight of sample after heating at 105°C (g)	TS (%)	MC ^{max} (%)	Average MC ^{max} (%)	STDEV	RSD (%)
Blotted-01	78.34	116	37.66	88.67	10.33	27.43	72.57	72.71	0.36	0.50
Blotted-02	82.38	122.76	40.38	93.51	11.13	27.56	72.44			
Blotted-03	68.67	96.61	27.94	76.18	7.51	26.88	73.12			
Unblotted-01	86.92	135.1	48.18	94.78	7.86	16.31	83.69	83.21	0.68	0.81
Unblotted-02	70.04	119.55	49.51	78.59	8.55	17.27	82.73			

Table B-2-6 Maximum moisture content (MC^{max}) of leaves

Sample	Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Weight of dish after heating at 105 °C (g)	Weight of sample after heating at 105°C (g)	TS (%)	Moisture (%)	Average moisture (%)	STDEV	RSD (%)
LV-1	24.25	42.02	17.77	27.66	3.41	19.19	80.81	80.78	0.50	0.62
LV-2	27.98	39.48	11.50	30.25	2.27	19.74	80.26			
LV-3	26.61	39.68	13.07	29.06	2.45	18.75	81.25			

Table B-2-7 Maximum moisture content (MC^{max}) of WC/BS mixtures

Sample	Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Weight of dish after heating at 105 °C (g)	Weight of sample after heating at 105°C (g)	TS (%)	Moisture (%)	Average moisture (%)	STDEV	RSD (%)
WC/BS-1	26.09	44.49	18.40	32.72	6.63	36.05	63.95	64.75	0.75	1.16
WC/BS-2	24.49	44.98	20.49	31.57	7.08	34.56	65.44			
WC/BS-3	31.55	51.03	19.48	38.40	6.85	35.15	64.85			

Note: [1] large amount of solid loss observed during the operation

[2] woodchips used in the mixture full-scale fine woodchips

Table B-3-1 Particle density of woodchips (6.35 < size < 12.70 mm)

Sample	Weight of empty flask W_p (g)	Weight of flask and sample (g)	Sample moisture content (%)	Weight of flask and sample corrected to oven dried weight W_s (g)	Weight of flask and sample, filled with boiled water W_{sw} (g)	Weight of flask and boiled water W_w (g)	Sample particle density PD (g/cm ³)	STDEV
1	243.6	282.4	31.56	270.16	1237.9	1229.8	1.44	0.26
2	226.4	264.5	31.56	252.48	1229.4	1218.9	1.67	
3	220.7	259.6	31.56	247.32	1226	1214.3	1.78	
4	241.7	281	31.56	268.60	1246.5	1234.1	1.85	
5	273.1	309.4	31.56	297.94	1263.6	1253.9	1.64	
6	246.3	288.5	31.56	275.18	1242.6	1229.3	1.85	
7	243.1	285.7	31.56	272.26	1242.3	1225.7	2.32	
8	242.7	284.7	31.56	271.45	1242.1	1226.4	2.20	
9	244.4	284.9	31.56	272.12	1242.8	1229	1.99	
10	241.2	287.9	31.56	273.16	1234.4	1221	1.72	
Temperature at which measurements were taken =				23.5	°C			
Density of water at which measurements were taken =				0.997314	g/cm ³			
PD = $p_w(W_s - W_p) / [(W_s - W_p) - (W_{sw} - W_w)]$								
Average PD =		1.84	g/cm ³					

Table B-3-2 Particle density of woodchips (0 - 19.05 mm)

Sample	Weight of empty flask W_p (g)	Weight of flask and sample (g)	Sample moisture content (%)	Weight of flask and sample corrected to oven dried weight W_s (g)	Weight of flask and sample, filled with boiled water W_{sw} (g)	Weight of flask and boiled water W_w (g)	Sample particle density PD (g/cm ³)	STDEV
1	243.7	282.9	29.31	271.41	1239.9	1229.9	1.56	0.08
2	226.3	264.6	29.31	253.37	1227.4	1219.4	1.42	
3	220.7	265.2	29.31	252.16	1228.1	1216.7	1.56	
Temperature at which measurements were taken =				22.5	°C			
Density of water at which measurements were taken =				0.99757	g/cm ³			
PD = $p_w(W_s - W_p) / [(W_s - W_p) - (W_{sw} - W_w)]$								
Average PD =		1.51	g/cm ³					

Table B-3-3 Particle density of straw

Sample	Weight of empty flask W_p (g)	Weight of flask and sample (g)	Sample moisture content (%)	Weight of flask and sample corrected to oven dried weight W_s (g)	Weight of flask and sample, filled with boiled water W_{sw} (g)	Weight of flask and boiled water W_w (g)	Sample particle density PD (g/cm ³)	STDEV
1	243.6	269.9	44.90	258.09	1234.8	1229.8	1.52	0.28
2	220.7	253.9	44.90	238.99	1220.4	1214.3	1.50	
3	241.7	267.8	44.90	256.08	1234.5	1234.1	1.03	
Average							1.35	
Temperature at which measurements were taken =				22.4	°C			
Density of water at which measurements were taken =				0.9975956	g/cm ³			
PD = $p_w(W_s - W_p) / [(W_s - W_p) - (W_{sw} - W_w)]$								

Table B-3-4 Particle density of wood shavings

Sample	Weight of empty flask W_p (g)	Weight of flask and sample (g)	Sample moisture content (%)	Weight of flask and sample corrected to oven dried weight W_s (g)	Weight of flask and sample, filled with boiled water W_{sw} (g)	Weight of flask and boiled water W_w (g)	Sample particle density PD (g/cm ³)	STDEV
1	243.6	273.5	10.28	270.43	1237.9	1229.8	1.43	0.16
2	241.7	273.5	10.28	270.23	1243.2	1234.1	1.46	
3	220.7	250.9	10.28	247.80	1225.7	1214.3	1.72	
Temperature at which measurements were taken =				23.5	°C			
Density of water at which measurements were taken =				0.997314	g/cm ³			
PD = $p_w(W_s - W_p) / [(W_s - W_p) - (W_{sw} - W_w)]$								
Average PD =		1.54	g/cm ³					

Table B-4-1 Bulk density of woodchips (6.35 < size < 12.70 mm)

Sample	Weight of biocell (g)	weight of biocell + sample (g)	Weight of Sample (g)	Fill Height (m)	Volume of sample (m3)	Bulk density (kg/m3)	Average Bulk Density (Kg/m3)
1	1.64	2.36	0.72	0.2	0.003627	198.49	197.11
2	1.65	2.36	0.71	0.2	0.003627	195.74	

Table B-4-2 Bulk density of woodchips (0 < size < 19.05 mm)

Sample	Weight of biocell (g)	weight of biocell + sample (g)	Weight of Sample (g)	Fill Height (m)	Volume of sample (m3)	Bulk density (kg/m3)	Average Bulk Density (Kg/m3)
1	1.65	2.53	0.88	0.2	0.003627	242.60	242.60
2	1.64	2.52	0.88	0.2	0.003627	242.60	

Table B-4-3 Bulk density of straw (unknown size)

Sample	Weight of biocell (g)	weight of biocell + sample (g)	Weight of Sample (g)	Fill Height (m)	Volume of sample (m3)	Bulk density (kg/m3)	Average Bulk Density (Kg/m3)
1	1.66	1.97	0.31	0.2	0.00363	85.46	86.84
2	1.65	1.97	0.32	0.2	0.00363	88.22	

Table B-4-4 Bulk density of wood shavings (unknown size)

Sample	Weight of biocell (g)	weight of biocell + sample (g)	Weight of Sample (g)	Fill Height (m)	Volume of sample (m3)	Bulk density (kg/m3)	Average Bulk Density (Kg/m3)
1	1.66	2.13	0.47	0.2	0.00363	129.57	126.82
2	1.65	2.1	0.45	0.2	0.00363	124.06	

Table B-5-1 TAS & FAS of woodchips (6.35 < size < 12.70 mm)

Part 1: Conditions of sample			
Bulk density =	197	kg/m ³	
Particle density =	1840	kg/m ³	
Moisture content =	30.98	%	
Maximum moisture content =	65.46	%	
Part 2: Porosity & TAS (at MC of 30.98 %)			
Porosity =	92.61	%	
Water content =	6.10	%	
Total air space (TAS) =	86.51	%	
Part 3: FAS (at maximum MC of 65.46 %)			
Assumed sample mass (m_t) =	100	kg	
Mass of water in original sample (m_w) =	30.98	kg	
Mass of water in saturated sample (m_w^{max}) =	130.81	kg	
Volume of air in original sample (V_a) =	0.10	m ³	
Volume of sample (V_t) =	0.51	m ³	
Volumetric ratio of air to sample (V_a/V_t) =	19.67	%	
Free air space (FAS) of sample =	66.84	%	

Table B-5-2 TAS & FAS of woodchips (0 < size < 19.05 mm)

Part 1: Conditions of sample			
Bulk density =	242	kg/m ³	
Particle density =	1510	kg/m ³	
Moisture content =	27.99	%	
Maximum moisture content =	63.61	%	
Part 2: Porosity & TAS (at MC of 28 %)			
Porosity =	88.46	%	
Water content =	6.77	%	
Total air space (TAS) =	81.69	%	
Part 3: FAS (at maximum MC of 63.61 %)			
Assumed sample mass (m_t) =	100	kg	
Mass of water in original sample (m_w) =	27.99	kg	
Mass of water in saturated sample (m_w^{max}) =	125.87	kg	
Volume of air in original sample (V_a) =	0.10	m ³	
Volume of sample (V_t) =	0.41	m ³	
Volumetric ratio of air to sample (V_a/V_t) =	23.69	%	
Free air space (FAS) of sample =	58.00	%	

Table B-5-3 TAS & FAS of straw

Part 1: Conditions of sample		
Bulk density =	87	kg/m ³
Particle density =	1350	kg/m ³
Moisture content =	32.5	%
Maximum moisture content =	83.45	%
Part 2: Porosity & TAS (at MC of 32.5 %)		
Porosity =	95.65	%
Water content =	2.83	%
Total air space (TAS) =	92.82	%
Part 3: FAS (at maximum MC of 83.45 %)		
Assumed sample mass (m_t) =	100	kg
Mass of water in original sample (m_w) =	32.5	kg
Mass of water in saturated sample (m_w^{\max}) =	340.35	kg
Volume of air in original sample (V_a) =	0.31	m ³
Volume of sample (V_t) =	1.15	m ³
Volumetric ratio of air to sample (V_a/V_t) =	26.78	%
Free air space (FAS) of sample =	66.04	%

Table B-5-4 TAS & FAS of wood shavings

Part 1: Conditions of sample		
Bulk density =	127	kg/m ³
Particle density =	1540	kg/m ³
Moisture content =	10.28	%
Maximum moisture content =	72.71	%
Part 2: Porosity & TAS (at MC of 32.5 %)		
Porosity =	92.60	%
Water content =	1.31	%
Total air space (TAS) =	91.30	%
Part 3: FAS (at maximum MC of 72.71 %)		
Assumed sample mass (m_t) =	100	kg
Mass of water in original sample (m_w) =	10.28	kg
Mass of water in saturated sample (m_w^{\max}) =	239.05	kg
Volume of air in original sample (V_a) =	0.23	m ³
Volume of sample (V_t) =	0.79	m ³
Volumetric ratio of air to sample (V_a/V_t) =	29.05	%
Free air space (FAS) of sample =	62.24	%

Table B-5-4 TAS & FAS of Leaves

Part 1: Conditions of sample		
Bulk density =	153.2	kg/m ³
Particle density =	1890	kg/m ³
Moisture content =	38.68	%
Maximum moisture content =	80.78	%
Part 2: Porosity & TAS (at MC of 32.5 %)		
Porosity =	95.03	%
Water content =	5.93	%
Total air space (TAS) =	89.10	%
Part 3: FAS (at maximum MC of 72.71 %)		
Assumed sample mass (m_t) =	100	kg
Mass of water in original sample (m_w) =	38.68	kg
Mass of water in saturated sample (m_w^{\max}) =	257.72	kg
Volume of air in original sample (V_a) =	0.22	m ³
Volume of sample (V_t) =	0.65	m ³
Volumetric ratio of air to sample (V_a/V_t) =	33.56	%
Free air space (FAS) of sample =	55.55	%

Note: Data of BD, PD and MC was cited from Gagne (2001)

Appendix C

Phase III Raw Data

Table C-1 Dry solids lost during the operation of MC^{max} of BS/WC (0 - 19.05 mm)

1. Solid lost by Draining				
Pan (g)	Pan+solids (g)	Solids lost (g)		
274.82	276.58	1.76		
274.82	277.36	2.54		
	Average =	2.15		
2. Solids lost by blotting				
Sponge (g)	Sponge+solids (g)	Solids lost (g)		
9.75	12.05	2.3		
9.77	12.21	2.44		
	Average =	2.37		
Sponge cloth (g)	Sponge cloth+solids (g)	Solids lost (g)		
10.05	12.84	2.79		
10.03	12.76	2.73		
	Average =	2.76		
3. Sample mass for max MC				
crucible (g)	crucible+sample (g)	crucible + dry sample (g)	dry sample (g)	MC ^{max} (%)
86.3	130.89	101.39	15.09	66.16
100.54	146.95	117.1	16.56	64.32
		Average =	15.83	65.24
4. Percentage of solid lost				
Total solids lost =		7.28 g		
Solids lost by blotting =		5.13 g		
Net sample mass =		15.83 g		
Total sample mass =		23.11 g		
Total percentage lost =		31.5%		
Percentage lost by blotting =		22.2%		

Table C-2-1 MC of woodchips/biosolids (size of woodchips: 0 - 19.05 mm)

Sample	Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Weight of dish after heating at 105 °C (g)	Weight of sample after heating at 105°C (g)	TS (%)	MC ^{max} (%)	Average MC ^{max} (%)	STDEV
1	68.67	141.87	73.2	101.59	32.92	44.97	55.03	55.58	0.52
2	70.85	141.09	70.24	101.99	31.14	44.33	55.67		
3	69.65	140.02	70.37	100.57	30.92	43.94	56.06		

Table C-2-2 MC of woodchips/biosolids (size of woodchips: 6.35 - 12.70 mm)

Sample	Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Weight of dish after heating at 105 °C (g)	Weight of sample after heating at 105°C (g)	TS (%)	MC ^{max} (%)	Average MC ^{max} (%)	STDEV
1	89.68	165.45	75.77	124.62	34.94	46.11	53.89	54.32	0.37
2	86.91	161.36	74.45	120.75	33.84	45.45	54.55		
3	84.29	164.22	79.93	120.64	36.35	45.48	54.52		

Table C-2-3 MC of straw/biosolids (size of straw: unknown)

Sample	Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Weight of dish after heating at 105 °C (g)	Weight of sample after heating at 105°C (g)	TS (%)	MC ^{max} (%)	Average MC ^{max} (%)	STDEV
1	90.86	136.54	45.68	110.54	19.68	43.08	56.92	56.98	0.06
2	91.92	139.6	47.68	112.43	20.51	43.02	56.98		
3	92.16	141.23	49.07	113.24	21.08	42.96	57.04		

Table C-3-1 Bulk density of biosolids

Sample	Weight of Dish (g)	Initial weight of dish + sample (g)	Weight of Initial Sample (g)	Volume of container (mL)	Bulk density (g/mL)	Average Bulk Density (g/mL)
WC-1	22.44	161.89	139.45	135	1.03	1.07
WC-2	22.38	169.76	147.38	135	1.09	
WC-3	23.22	168.18	144.96	135	1.07	

Table C-3-2 Bulk density of BS/WC (size of woodchips: 0 - 19.05 mm)

Sample	Weight of biocell (g)	weight of biocell + sample (g)	Weight of Sample (g)	Fill Height (m)	Volume of sample (m3)	Bulk density (kg/m ³)	Average Bulk Density (Kg/m ³)	STDEV	RSD (%)
1	1.66	3.35	1.69	0.153	0.00277	609.03	610.83	2.55	0.42
2	1.65	3.35	1.7	0.153	0.00277	612.63			

Table C-3-3 Bulk density of BS/WC (size of woodchips: 6.35 - 12.70 mm)

Sample	Weight of biocell (g)	weight of biocell + sample (g)	Weight of Sample (g)	Fill Height (m)	Volume of sample (m3)	Bulk density (kg/m ³)	Average Bulk Density (Kg/m ³)	STDEV	RSD (%)
1	1.66	3.36	1.7	0.193	0.0035	485.66	484.23	2.02	0.42
2	1.65	3.34	1.69	0.193	0.0035	482.81			

Table C-3-4 Bulk density of BS/straw (size of straw: unknown)

Sample	Weight of biocell (g)	weight of biocell + sample (g)	Weight of Sample (g)	Fill Height (m)	Volume of sample (m3)	Bulk density (kg/m ³)	Average Bulk Density (Kg/m ³)	STDEV	RSD (%)
1	1.66	2.62	0.96	0.153	0.00277	345.96	349.56	5.10	1.46
2	1.65	2.63	0.98	0.153	0.00277	353.17			

Table C-4-1 Particle density of woodchips/biosolids (size of woodchips: 0 - 19.05 mm)

Sample	Weight of empty flask W_p (g)	Weight of flask and sample (g)	Sample moisture content (%)	Weight of flask and sample corrected to oven dried weight W_s (g)	Weight of flask and sample, filled with boiled water W_{sw} (g)	Weight of flask and boiled water W_w (g)	Sample particle density PD (g/cm ³)	STDEV
1	234.6	280.2	55.58	254.86	1234.3	1224.5	1.93	0.06
2	244.3	284.6	55.58	262.20	1241.2	1233.2	1.80	
3	246.3	288.6	55.58	265.09	1242.5	1233.8	1.86	
Temperature				25.4	°C			
Density of water at the above temperature =				0.9968276	g/cm ³			
$PD = p_w(W_s - W_p) / [(W_s - W_p) - (W_{sw} - W_w)]$								
Average PD =		1.86	g/cm ³					

Table C-4-2 Particle density of woodchips/biosolids (size of woodchips: 6.35 - 12.70 mm)

Sample	Weight of empty flask W_p (g)	Weight of flask and sample (g)	Sample moisture content (%)	Weight of flask and sample corrected to oven dried weight W_s (g)	Weight of flask and sample, filled with boiled water W_{sw} (g)	Weight of flask and boiled water W_w (g)	Sample particle density PD (g/cm ³)	STDEV
1	243.1	268.5	54.32	254.70	1234.8	1229.6	1.81	0.09
2	242.7	275.2	54.32	257.55	1241.5	1234.3	1.94	
3	244.4	276.5	54.32	259.06	1237.6	1231.2	1.77	
Temperature				22.5	°C			
Density of water at the above temperature =				0.99757	g/cm ³			
$PD = p_w(W_s - W_p) / [(W_s - W_p) - (W_{sw} - W_w)]$								
Average PD =		1.84	g/cm ³					

Table C-4-3 Particle density of straw/biosolids (size of straw: unknown)

Sample	Weight of empty flask W_p (g)	Weight of flask and sample (g)	Sample moisture content (%)	Weight of flask and sample corrected to oven dried weight W_s (g)	Weight of flask and sample, filled with boiled water W_{sw} (g)	Weight of flask and boiled water W_w (g)	Sample particle density PD (g/cm ³)	STDEV
1	243.6	267.5	56.98	253.88	1233.7	1229.5	1.69	0.03
2	242.5	272.6	56.98	255.45	1238.3	1233.2	1.65	
3	242.7	274.8	56.98	256.51	1226.8	1221.5	1.62	
Temperature				23.5	°C			
Density of water at the above temperature =				0.997314	g/cm ³			
PD = $p_w(W_s - W_p) / [(W_s - W_p) - (W_{sw} - W_w)]$								
Average PD =		1.65	g/cm ³					

Table C-5-1 TAS woodchips/biosolids (size of woodchips: 0 - 19.05 mm)

Part 1: Conditions of sample			
Bulk density =	610.83	kg/m ³	
Particle density =	1860	kg/m ³	
Moisture content =	55.58	%	
Part 2: Porosity & TAS (at MC of 28 %)			
Porosity =	85.41	%	
Water content =	33.95	%	
Total air space (TAS) =	51.46	%	

Table C-5-2 TAS woodchips/biosolids (size of woodchips: 6.35 - 12.70 mm)

Part 1: Conditions of sample			
Bulk density =	484.23	kg/m ³	
Particle density =	1840	kg/m ³	
Moisture content =	54.32	%	
Part 2: Porosity & TAS (at MC of 28 %)			
Porosity =	87.98	%	
Water content =	26.30	%	
Total air space (TAS) =	61.68	%	

Table C-5-3 TAS straw/biosolids (size of straw: unknown)

Part 1: Conditions of sample			
Bulk density =	349.56	kg/m ³	
Particle density =	1650	kg/m ³	
Moisture content =	56.98	%	
Part 2: Porosity & TAS (at MC of 28 %)			
Porosity =	90.89	%	
Water content =	19.92	%	
Total air space (TAS) =	70.97	%	

Table C-6-1 FAS of woodchips/biosolids (size of woodchips: 0 - 19.05 mm)

Part 1:		Conditions of raw materials & mixture				
Materials		Mass (Kg)	BD Kg/L	Volume (L)	MC (%)	Solids (%)
Substrate (s)		1.23	1.07	1.15	71.95	28.05
Bulking agent (b)		0.77	0.242	3.18	27.99	72.01
Mixture (m)	Sample #1	2	0.609	3.28	55.58	44.42
	Sample #2	2	0.613	3.26	55.58	44.42
Part 2:		Calculation Model				
$FAS_m = 1 - \frac{1}{R_{mb}} \left[\frac{1}{R_{bs}} + (1 - FAS_b) - \left(\frac{BD_b}{\rho_w} \right) \left(\frac{S_b}{S_m} - 1 \right) \right]$						
Part 3:		Parameters				
		Sample #1	Sample #2			
$R_{bs} =$	$V_b / V_s =$	2.77	2.77			
$R_{mb} =$	$V_m / V_b =$	1.03	1.03			
	$BD_b =$	0.242		kg/L		
	$\rho_w =$	1		kg/L		
	$S_s =$	28.05		%		
	$S_m =$	44.42		%		
	$FAS_b =$	0.5898				
Part 4:		FAS of Mixture				
	Sample #1	Sample #2	Average	STDEV	RSD (%)	
$FAS_m =$	0.3982	0.3942	0.3962	0.0028	0.71	

Table C-6-2 FAS of woodchips/biosolids (size of woodchips: 6.35 - 12.70 mm)

Part 1:		Conditions of raw materials & mixture				
Materials		Mass (Kg)	BD Kg/L	Volume (L)	MC (%)	Solids (%)
Substrate (s)		1.17	1.07	1.09	71.95	28.05
Bulking agent (b)		0.83	0.197	4.21	31.56	68.44
Mixture (m)	Sample #1	2	0.483	4.14	54.32	45.68
	Sample #2	2	0.486	4.12	54.32	45.68
Part 2:		Calculation Model				
$FAS_m = 1 - \frac{1}{R_{mb}} \left[\frac{1}{R_{bs}} + (1 - FAS_b) - \left(\frac{BD_b}{\rho_w} \right) \left(\frac{S_b}{S_m} - 1 \right) \right]$						
Part 3:		Parameters				
		Sample #1	Sample #2			
$R_{bs} =$	$V_b / V_s =$	3.85	3.85			
$R_{mb} =$	$V_m / V_b =$	0.98	0.98			
	$BD_b =$	0.197		kg/L		
	$\rho_w =$	1		kg/L		
	$S_s =$	28.05		%		
	$S_m =$	45.68		%		
	$FAS_b =$	0.6573				
Part 4:		FAS of Mixture				
	Sample #1	Sample #2	Average	STDEV	RSD (%)	
$FAS_m =$	0.4871	0.4839	0.4855	0.0023	0.46	

Table C-6-3 FAS of straw/biosolids (size of straw: unknown)

Part 1: Conditions of raw materials & mixture						
Materials		Mass (Kg)	BD Kg/L	Volume (L)	MC (%)	Solids (%)
Substrate (s)		1.14	1.07	1.07	71.95	28.05
Bulking agent (b)		0.86	0.08684	9.90	32.5	67.5
Mixture (m)	Sample #1	2	0.346	5.78	56.98	43.02
	Sample #2	2	0.353	5.67	56.98	43.02
Part 2: Calculation Model						
$FAS_m = 1 - \frac{1}{R_{mb}} \left[\frac{1}{R_{bs}} + (1 - FAS_b) - \left(\frac{BD_b}{\rho_w} \right) \left(\frac{S_b}{S_m} - 1 \right) \right]$						
Part 3: Parameters		Sample #1	Sample #2			
$R_{bs} =$	$V_b / V_s =$	9.30	9.30			
$R_{mb} =$	$V_m / V_b =$	0.58	0.57			
	$BD_b =$	0.08684		kg/L		
	$\rho_w =$	1		kg/L		
	$S_s =$	67.5		%		
	$S_m =$	43.02		%		
	$FAS_b =$	0.6625				
Part 4: FAS of Mixture						
	Sample #1	Sample #2	Average	STDEV	RSD (%)	
$FAS_m =$	0.3221	0.3084	0.3153	0.0097	3.08	

Appendix D:

Statistical Reports

Statistical Report: D-1

Sunday, June 01, 2003, 12:09:33

t-test (for BD of full-scale coarse woodchips (< 50.08 mm) and full-scale fine woodchips (< 19.05 mm))

Data source: Table A-3-1, A-3-2, and data from Eftoda and McCartney (2002)

Normality Test: Passed (P = 0.756)

Equal Variance Test: Passed (P = 0.072)

Group	N	Missing
Col 1	3	1
Col 2	5	1

Group	Mean	Std Dev	SEM
Col 1	0.125	0.00424	0.00300
Col 2	0.162	0.0117	0.00584

Difference -0.0375

t = -4.184 with 4 degrees of freedom. (P = 0.014)

95 percent confidence interval for difference of means: -0.0623 to -0.0126

The difference in the mean values of the two groups is greater than would be expected by chance; there is a statistically significant difference between the input groups (P = 0.014).

Power of performed test with alpha = 0.050: 0.859

Statistical Report: D-2

Sunday, June 01, 2003, 12:10:37

t-test (for BD of full-scale fine woodchips (0 – 19.05 mm) and sieved full-scale fine woodchips (3.18 - 9.53 mm))

Data source: Table A-3-1, A-3-2, and data from Eftoda and McCartney (2002)

Normality Test: Passed ($P = 0.752$)

Equal Variance Test: Passed ($P = 0.064$)

Group	N	Missing
Col 2	5	1
Col 3	3	1

Group	Mean	Std Dev	SEM
Col 2	0.162	0.0117	0.00584
Col 3	0.0977	0.00382	0.00270

Difference 0.0643

$t = 7.205$ with 4 degrees of freedom. ($P = 0.002$)

95 percent confidence interval for difference of means: 0.0395 to 0.0890

The difference in the mean values of the two groups is greater than would be expected by chance; there is a statistically significant difference between the input groups ($P = 0.002$).

Power of performed test with $\alpha = 0.050$: 0.999

Dry BD of woodchips:

Coarse woodchips (0 - 50.8 mm)	Fine woodchips (0-19.05 mm)	Sieved fine woodchips (3.18 – 9.53 mm)
0.1215	0.1687	0.095
0.1275	0.1745	0.1004
	0.1490	
	0.1557	

Statistical Report: D-3

Sunday, June 01, 2003, 12:37:59

One Way Analysis of Variance (for PD of all four kinds of woodchips)

Data source: Table A-5-1, A-5-2, A-5-3 and data from Eftoda and McCartney (2002)

Normality Test: Passed (P = 0.163)

Equal Variance Test: Passed (P = 0.278)

Group	N	Missing
Col 1	3	1
Col 3	4	1
Col 2	4	1
Col 4	5	1

Group	Mean	Std Dev	SEM
Col 1	1.740	0.170	0.120
Col 3	1.543	0.112	0.0644
Col 2	1.657	0.0513	0.0296
Col 4	1.805	0.348	0.174

Power of performed test with alpha = 0.050: 0.050

The power of the performed test (0.050) is below the desired power of 0.800.
You should interpret the negative findings cautiously.

Source of Variation	DF	SS	MS	F	P
Between Treatments	3	0.126	0.0419	0.795	0.530
Residual	8	0.422	0.0527		
Total	11	0.548			

The differences in the mean values among the treatment groups are not great enough to exclude the possibility that the difference is due to random sampling variability; there is not a statistically significant difference (P = 0.530).

Statistical Report: D-4

Saturday, July 12, 2003, 12:48:02

t-test (for FAS of full-scale coarse and lab-scale woodchips)

Data source: Table A-6-3 and A-6-4

Normality Test: Passed ($P = 0.621$)

Equal Variance Test: Passed ($P = 0.291$)

Group	N	Missing
Col 1	2	0
Col 2	4	0

Group	Mean	Std Dev	SEM
Col 1	89.430	0.707	0.500
Col 2	82.910	1.460	0.730

Difference 6.520

$t = 5.734$ with 4 degrees of freedom. ($P = 0.005$)

95 percent confidence interval for difference of means: 3.363 to 9.677

The difference in the mean values of the two groups is greater than would be expected by chance; there is a statistically significant difference between the input groups ($P = 0.005$).

Power of performed test with $\alpha = 0.050$: 0.985

Statistical Report: D-5

Friday, June 06, 2003, 10:48:01

t-test (for porosities measured by TMECC and PD method)

Data source: Table 4-3

Normality Test: Passed ($P = 0.073$)

Equal Variance Test: Passed ($P = 0.303$)

Group	N	Missing
Col 1	5	1
Col 2	5	1

Group	Mean	Std Dev	SEM
Col 1	65.922	16.130	8.065
Col 2	92.483	2.527	1.263

Difference -26.560

$t = -3.254$ with 6 degrees of freedom. ($P = 0.017$)

95 percent confidence interval for difference of means: -46.535 to -6.585

The difference in the mean values of the two groups is greater than would be expected by chance; there is a statistically significant difference between the input groups ($P = 0.017$).

Power of performed test with $\alpha = 0.050$: 0.739

The power of the performed test (0.739) is below the desired power of 0.800.
You should interpret the negative findings cautiously.

Statistical Report: D-6

Friday, June 06, 2003, 10:54:24

t-test (for FAS measured by TMECC and PD method)

Data source: Table 4-3

Normality Test: Passed ($P = 0.326$)

Equal Variance Test: Passed ($P = 0.176$)

Group	N	Missing
Col 3	5	1
Col 4	5	1

Group	Mean	Std Dev	SEM
Col 3	32.410	15.774	7.887
Col 4	80.695	9.245	4.622

Difference -48.285

$t = -5.282$ with 6 degrees of freedom. ($P = 0.002$)

95 percent confidence interval for difference of means: -70.654 to -25.916

The difference in the mean values of the two groups is greater than would be expected by chance; there is a statistically significant difference between the input groups ($P = 0.002$).

Power of performed test with $\alpha = 0.050$: 0.992

Statistical Report: D-7

Sunday, April 13, 2003, 01:35:35

t-test (for MC^{max} of woodchips by first and second operators)

Data source: Table B-2-1 and B-2-2

Normality Test: Passed (P = 0.842)

Equal Variance Test: Failed (P = 0.020)

Group	N	Missing
Col 12	11	1
Col 13	4	1

Group	Mean	Std Dev	SEM
Col 12	65.457	0.724	0.229
Col 13	64.697	1.354	0.781

Difference 0.760

t = 1.323 with 11 degrees of freedom. (P = 0.213)

95 percent confidence interval for difference of means: -0.505 to 2.025

The difference in the mean values of the two groups is not great enough to reject the possibility that the difference is due to random sampling variability. There is not a statistically significant difference between the input groups (P = 0.213).

Power of performed test with alpha = 0.050: 0.118

The power of the performed test (0.118) is below the desired power of 0.800. You should interpret the negative findings cautiously.

Statistical Report: D-8

Saturday, April 12, 2003, 23:48:48

t-test (for MC^{max} between screened and unscreened full-scale fine woodchips)

Data source: Table B-2-3

Normality Test: Passed (P = 0.685)

Equal Variance Test: Passed (P = 0.974)

Group	N	Missing
Col 1	4	1
Col 2	4	1

Group	Mean	Std Dev	SEM
Col 1	64.107	0.998	0.576
Col 2	63.613	1.000	0.578

Difference 0.493

t = 0.605 with 4 degrees of freedom. (P = 0.578)

95 percent confidence interval for difference of means: -1.771 to 2.758

The difference in the mean values of the two groups is not great enough to reject the possibility that the difference is due to random sampling variability. There is not a statistically significant difference between the input groups (P = 0.578).

Power of performed test with alpha = 0.050: 0.050

The power of the performed test (0.050) is below the desired power of 0.800. You should interpret the negative findings cautiously.

Statistical Report: D-9

Sunday, April 13, 2003, 00:27:32

One Way Analysis of Variance (for MC^{max} of full-scale fine woodchips caused by different draining time: 5, 15, 30, and 60 minutes)

Data source: Table B-2-3

Normality Test: Passed (P = 0.328)

Equal Variance Test: Passed (P = 0.361)

Group	N	Missing
Col 3	4	1
Col 4	4	1
Col 5	4	1
Col 6	4	1

Group	Mean	Std Dev	SEM
Col 3	64.107	0.998	0.576
Col 4	64.600	0.844	0.487
Col 5	64.387	0.506	0.292
Col 6	63.393	0.110	0.0636

Power of performed test with alpha = 0.050: 0.136

The power of the performed test (0.136) is below the desired power of 0.800.
You should interpret the negative findings cautiously.

Source of Variation	DF	SS	MS	F	P
Between Treatments	3	2.489	0.830	1.679	0.248
Residual	8	3.953	0.494		
Total	11	6.443			

The differences in the mean values among the treatment groups are not great enough to exclude the possibility that the difference is due to random sampling variability; there is not a statistically significant difference (P = 0.248).

Statistical Report: D-10

Sunday, April 13, 2003, 01:20:15

t-test (for MC^{max} of blotted and un-blotted full-scale fine woodchips)

Data source: Table B-2-3

Normality Test: Passed (P = 0.089)

Equal Variance Test: Failed (P = 0.023)

Group	N	Missing
Col 7	4	1
Col 8	4	1

Group	Mean	Std Dev	SEM
Col 7	64.107	0.998	0.576
Col 8	70.773	0.0839	0.0484

Difference -6.667

t = -11.534 with 4 degrees of freedom. (P = <0.001)

95 percent confidence interval for difference of means: -8.271 to -5.062

The difference in the mean values of the two groups is greater than would be expected by chance; there is a statistically significant difference between the input groups (P = <0.001).

Power of performed test with alpha = 0.050: 1.000

Statistical Report: D-11

Sunday, April 13, 2003, 01:30:57

t-test (for MC^{max} of blotted and un-blotted wood shavings)

Data source: Table B-2-5

Normality Test: Passed (P = 0.539)

Equal Variance Test: Passed (P = 0.574)

Group	N	Missing
Col 9	4	1
Col 10	3	1

Group	Mean	Std Dev	SEM
Col 9	72.710	0.361	0.208
Col 10	83.210	0.679	0.480

Difference -10.500

t = -23.456 with 3 degrees of freedom. (P = <0.001)

95 percent confidence interval for difference of means: -11.925 to -9.075

The difference in the mean values of the two groups is greater than would be expected by chance; there is a statistically significant difference between the input groups (P = <0.001).

Power of performed test with alpha = 0.050: 1.000

Statistical Report: D-12

Saturday, June 21, 2003, 18:58:10

t-test (for FAS determined by the new numerical model and PD method for composting mixtures)

Data source: Table 4-10

Normality Test: Passed ($P = 0.723$)

Equal Variance Test: Passed ($P = 0.231$)

Group	N	Missing
Col 1	3	0
Col 2	3	0

Group	Mean	Std Dev	SEM
Col 1	39.900	8.513	4.915
Col 2	61.340	9.807	5.662

Difference -21.440

$t = -2.860$ with 4 degrees of freedom. ($P = 0.046$)

95 percent confidence interval for difference of means: -42.257 to -0.623

The difference in the mean values of the two groups is greater than would be expected by chance; there is a statistically significant difference between the input groups ($P = 0.046$).

Power of performed test with $\alpha = 0.050$: 0.528

The power of the performed test (0.528) is below the desired power of 0.800. You should interpret the negative findings cautiously.