Compostability of municipal and industrial wastewater sludge: physical, chemical and biochemical transformations and end-product characteristics

by

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Table of contents

ii

List of figures

iv

v

vi

List of tables

Abbreviations

ACT-IND	activated industrial (sludge)
ACT-MUN	activated municipal (sludge)
BVS	biodegradable volatile solids
CEC	cation exchange capacity
DIG-MUN	digested municipal (sludge)
EC	electrical conductivity
EXC	alkali extractable carbon
LOIOM	loss on ignition organic matter (synonymous to VS)
OM	organic matter
TOC	total organic carbon
VS	volatile solids (synonymous to LOIOM)
WHC	water holding capacity
WWS	wastewater sludge

Abstract

Can composting save the world from its human generated biological waste problem and give a diversifying organic agriculture a much needed boost at the same time? The age old natural process of composting has been well researched and documented in scientific and other literature. Thermophilic composting transforms biological/organic waste into a stable plant and soil friendly material (compost) that has been sanitised through high temperature and humified at the same time – a true miracle of nature.

Mixtures for thermophilic composting can be formulated fairly accurately from their water, C and N content. The pH needs to be within an acceptable range (5-8) but can be adjusted with agricultural lime if needed. The EC and ash content of sludge, together with the C and N content, are fairly reliable indicators of available bio-chemical energy for composting. Monitoring the temperature and water content gives sufficient feedback for proper management of the process. Bio-chemical transformations in composting material tell the tale of the microbial processes at the heart of composting and humification. The quality of compost depends largely on the quality of the raw materials used, even if mixture formulation and process management is effective.

The study firstly evaluated the compostability of various types of WWS and eucalyptus sawdust bulking agent with the focus on the known requirements and conditions for thermophilic aerobic composting. This entailed a composting experiment during which the sludge and bulking agent were characterised, feedstock mixtures formulated and certain composting conditions (temperature and water content) and feedstock characteristics (pH, EC and ash content) monitored over time as piles were managed through turning, sampling and watering.

Secondly, the study evaluated the quality of compost produced from various types of WWS and eucalyptus sawdust bulking agent as influenced by the raw material and initial feedstock characteristics, microbial dynamics and environmental conditions during composting and as indicated by certain chemical and biochemical transformations

(including OM decomposition) during composting and end product (compost) characteristics.

Tracking the chemical and biochemical evolution during composting in the four treatments entailed the laboratory analyses of a variety of signature characteristics for samples taken at the start of composting, at the end of the thermophilic phase when pile temperature decreased to below 40ºC and at the end of the curing phase of composting when the temperature of piles did not exceed ambient temperature. Finally, a seedling emergence bioassay was performed as a direct test of compost maturity and quality.

The main findings of this study are that dewatered, activated sludge has a high level of thermophilic compostability with Eucalyptus sawdust as bulking agent. Dried, digested sludge has a low level of compostability with Eucalyptus sawdust as bulking agent mainly because of the anaerobic digestion that depleted the C and bio-chemical energy in the sludge. Little value is added to digested sludge through composting with eycalyptus sawdust except that it probably facilitated a further "curing" of the already stable material.

Overall, dewatered ACT-MUN sludge and eucalyptus sawdust produced the compost of highest quality and stability/maturity in a volume ratio of 1:4 (sludge:sawdust). Dewatered ACT-IND sludge and eucalyptus sawdust produced an interesting compost of high stability/maturity with a high level of humification and medium fertility status. Increasing the sludge:sawdust ratio closer to 1:4 (volume) will enhance the chemical quality of this good compost. Dried DIG-MUN sludge and eucalyptus sawdust produced a stable compost of very low pH, relatively low fertility, poor physical character and high risk of phytotoxicity.

Composting is a great option for WWS treatment that deserves a lot more attention in South Africa.

Introduction

Human generated biological waste and the management thereof, covers the whole spectrum of society - from first world wastewater treatment plants to third world informal settlements. Large quantities of domestic and industrial wastewater sludge (WWS) are generated daily by wastewater treatment plants across South Africa. Simultaneously, thousands of formal and informal households in South Africa are still operating with sub basic sanitation such as the well-known "long drop" or often without any sanitation system at all.

Historically, lack of external nutrient sources as well as challenges with soil degradation in the agricultural sector, forced people to care about the cycling of organic materials (Insam, Riddech and Klammer, 2002). In traditional mixed farming systems, soils were amended with compost from plant residues and animal manures for thousands of years, killing two birds with one stone: nutrient supply and long-term soil sustainability. Today, specialised high-yielding mono-crop farming systems are driven by chemical fertilisation. Free market global trade, ever increasing population growth and urbanisation in many countries, contribute generously towards a society that has forgotten the importance of traditional recycling. Furthermore, the economic value regarding recycling is disputed. Centralised dumps or incineration plants are often the simple answer to growing waste problems (Insam *et al*., 2002).

The increasing pollution pressure on soil and water resources and the need for responsible, environmentally sound and sustainable waste recycling/re-use programmes has prompted serious attention. East Rand Water Care Works (ERWAT) and petro-chemical giant SASOL entered into a research project in collaboration with the Department of Plant Production and Soil Science - University of Pretoria - to investigate and propose various sustainable strategies for the beneficial utilization of WWS in plant production and soil management. A variety of WWS types are produced from waste water treatment plants. Two major types are (1) waste activated sludge and (2) anaerobically digested sludge. Digested sludge is generally more stable and contains less

1

biodegradable volatile solids (BVS) than activated sludge, but the degree of stability varies considerably with specific process conditions at different treatment plants (Snyman and Herselman, 2006).

WWS is predominantly organic material with a high carbon content that contains plant nutrients (nitrogen, potassium, phosphorus, calcium and micronutrients) and can be utilised as a soil conditioner and fertiliser by either direct application to soil, or applied as sludge compost. "Compost is an organic material that has been stabilised to a humus-like product, that is free of viable human and plant pathogens and plant seeds, that does not attract insects or vectors, that can be handled and stored without nuisance, and that is beneficial to the growth of plants" (Haug, 1993). Apart from soil application, compost can also be used as mulch, top dressing and as a component of container growing media (Haug, 1993).

Generally composting of organic waste is seen as a very good method of waste stabilisation with considerable environmental advantages compared to landfills, sacrificial lands and other conventional disposal methods for the decomposition of solid domestic wastes and this practice is likely to increase in future (Atlas and Bartha, 1998). Major reasons for non-practice are economic viability, odours, flies and in the case of WWS, pathogenic and chemical safety (heavy metal content).

The study firstly evaluated the compostability of various types of WWS and eucalyptus sawdust bulking agent with the focus on the known requirements and conditions for thermophilic aerobic composting. This entailed a composting experiment during which the sludge and bulking agent were characterised, feedstock mixtures formulated and certain composting conditions (temperature and water content) and feedstock characteristics (pH, EC and ash content) monitored over time as piles were managed through turning, sampling and watering.

It is proposed that the compostability of different types of WWS and eucalyptus sawdust bulking agent can be evaluated if a physically and chemically conditioned feedstock is

formulated and the composting process conditions appropriately managed through turning and watering while monitoring and evaluating certain composting conditions and feedstock characteristics during composting.

Some of the important process/environmental conditions include degree of aeration (oxygen availability), temperature, water content, air filled porosity and other substrate characteristics like pH, carbon to nitrogen ratio (C:N) and matrix structure (Cunha Queda *et al*., 2002).

Secondly, the study evaluated the quality of compost produced from various types of WWS and eucalyptus sawdust bulking agent as influenced by the raw material and initial feedstock characteristics, microbial dynamics and environmental conditions during composting and as indicated by certain chemical and biochemical transformations (including OM decomposition) during composting and end product (compost) characteristics.

The temporal changes in certain chemical and biochemical characteristics observed in composting materials, give insight into the basic processes that constitute composting. These observations also provide insightful parameters for the evaluation of the suitability of raw materials and feedstock for composting, systematic monitoring as well the evaluation of compost quality. This study postulates that compost produced from different WWS and Eucalyptus sawdust (bulking agent) can be expected to be of differing qualities.

Knowledge and understanding of the various transformations (initial substrate characteristics as well as composting environment influences) together with the total impact of this on the characteristics and quality of the compost should be applied to optimise the composting process and compost functionality in various plant production and soil management applications. Any predictive capability and true control of the composting process and compost quality can only arise with a good knowledge of certain

chemical and physical characteristics of individual materials and the composting feedstock as a whole.

This study does not address the bioavailability of nutrients from compost or the pathogenic and chemical safety of compost directly (except where these are referred to in the literature review).

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Chapter 1: Principles of successful composting – a review

1.1   Introduction

Haug (1993) defines composting as *the biological decomposition and stabilisation of organic substrates, under conditions that allow development of thermophillic temperatures as a result of biologically produced heat, to produce a final product that is stable, free of pathogens and plant seeds, and can be beneficially applied to land.* Composting here refers to the aerobic decomposition of organic materials which requires oxygen and produces carbon dioxide, water and heat.

The aim of this literature study is to present the underlying principles and required conditions for composting of organic substrates with special reference to the composting of municipal and industrial WWS.

1.2   Composting process principles

Probably all of the principles involved in composting organic wastes can be traced to a single all-inclusive fact, that composting is a biological process. Composting therefore, has the limitations of biological systems and the process is affected by the basic environmental conditions that influence all biological activity. The ultimate limitation stemming from the biological nature of the composting process is the genetic make-up of the organisms involved. This implies that a definite upper limit exists with respect to the rate and extent of the composting process, beyond which sophistication of equipment and operation and enhancement of environmental conditions, cannot be of any further use. The proper environmental conditions are, however, a requisite to the realisation of the genetic potential of the organisms (Golueke, 1982a).

1.3   Conditions for composting

Both mesophilic and thermophilic micro-organisms contribute to composting and the process is most rapid when conditions that encourage their growth are established and maintained. Such conditions require a mixture of materials (feedstock) that provide the nutrients needed for microbial activity and growth, including a balanced supply of available carbon and nitrogen, oxygen to keep the process aerobic, as well as enough

water to permit biological activity without hindering aeration (Cundiff and Mankin, 2003).

Composting occurs to various degrees over a wide range of conditions. The end use or desired quality of the compost dictates the amount of control that must be maintained during the process. Recommended conditions for effective composting are given in Table 1.1.

Table 1.1 Recommended conditions for rapid composting (Cundiff and Mankin, 2003)

Condition	Reasonable range	Preferred range for optimal (rapid) composting
C:N ratio	20:1 – 40:1	25:1 – 30:1
Water content (% of wet mass)	40 – 65%	50 – 60%
Oxygen concentrations (mole fraction)	> 5%	>> 5%
Particle size (diameter, mm)	5 – 15	Varies
pH	5.5 – 9.0	6.5 – 8.0
Temperature of active phase (°C)	40 – 65	55 – 60

1.4 Aeration, temperature and microbial dynamics

Composting is accomplished in static piles/windrows, aerated piles/windrows or continuous feed reactors. Although generally defined as an aerobic process, anaerobic zones often occur in a compost pile where mass transfer is limited by sub-optimal porosity, excessive water content or because of a high rate of decomposition producing excessive CO_2. Oxygen concentrations in a compost pile are usually five times lower than in ambient air (Figure 1.1), even when mechanically turned (Atlas and Bartha, 1998).

7
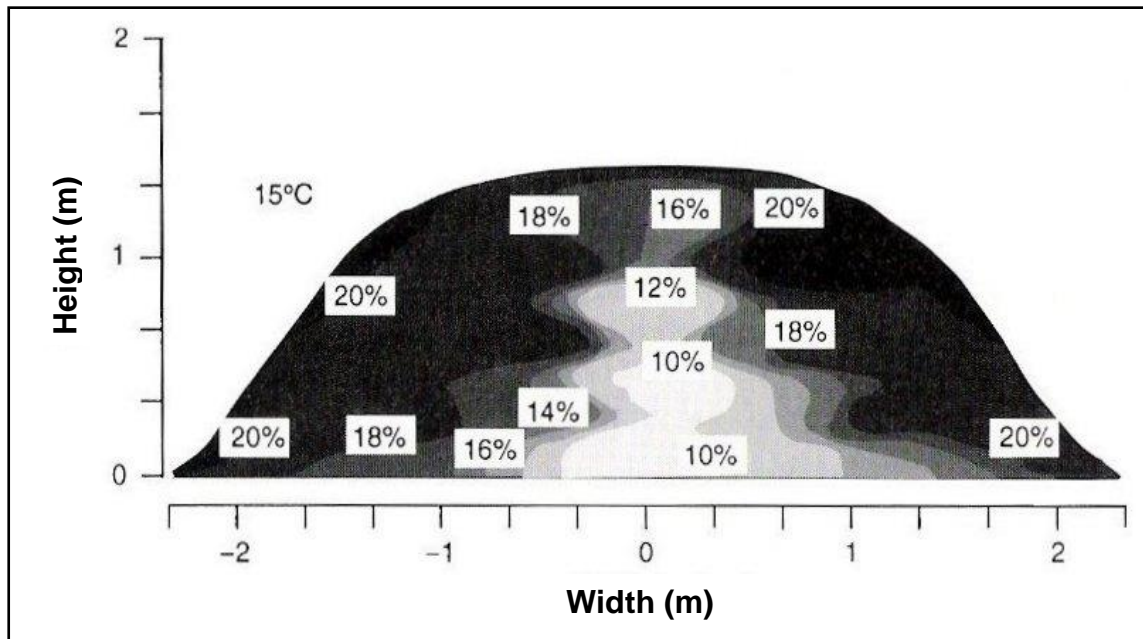
Figure 1.1 Typical concentration of oxygen in a compost pile/windrow shown as percent of oxygen concentration of ambient air at 15°C (Diaz *et al*., 1993)

During static pile composting, natural draught/convection ventilation occurs as a result of the density difference between moist, warm air (gases) within the windrow or pile and cooler, drier, ambient air. The rate of ventilation is a function of this air density difference (which will increase with higher internal pile temperature), particle size and free air space of the composting mixture. Mechanical turning serves to re-establish the free air space and achieve the maximum possible ventilation rate for a particular composting mixture (Haug 1993).

Composting is a dynamic process carried out by a rapid succession of mixed microbial populations. The microorganisms transform organic matter into CO_2, microbial biomass, heat and a humus-like end product (compost). The precise nature of succession and the number of micro-organisms at each composting phase is dependent on the substrate and on the preceding microorganisms in the succession (Crawford, 1983 in Tuomela *et al.*, 2000).

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The process starts at ambient temperature, is initiated by mesophilic heterotrophs and initially mesophilic bacteria predominate. The soluble and easily degradable carbon sources, such as monosaccharides, starch and lipids are utilised by the micro-organisms and the aerobic oxidation reactions catalysed by these micro-organisms produce heat (McKinley and Vestal, 1985; Crawford, 1983 in Tuomela *et al.*, 2000). Under favourable conditions, this self-heating in static piles/windrows typically raises the temperature inside a compost pile/windrow to 55-60°C or above, within two to three days (Figure 1.2).
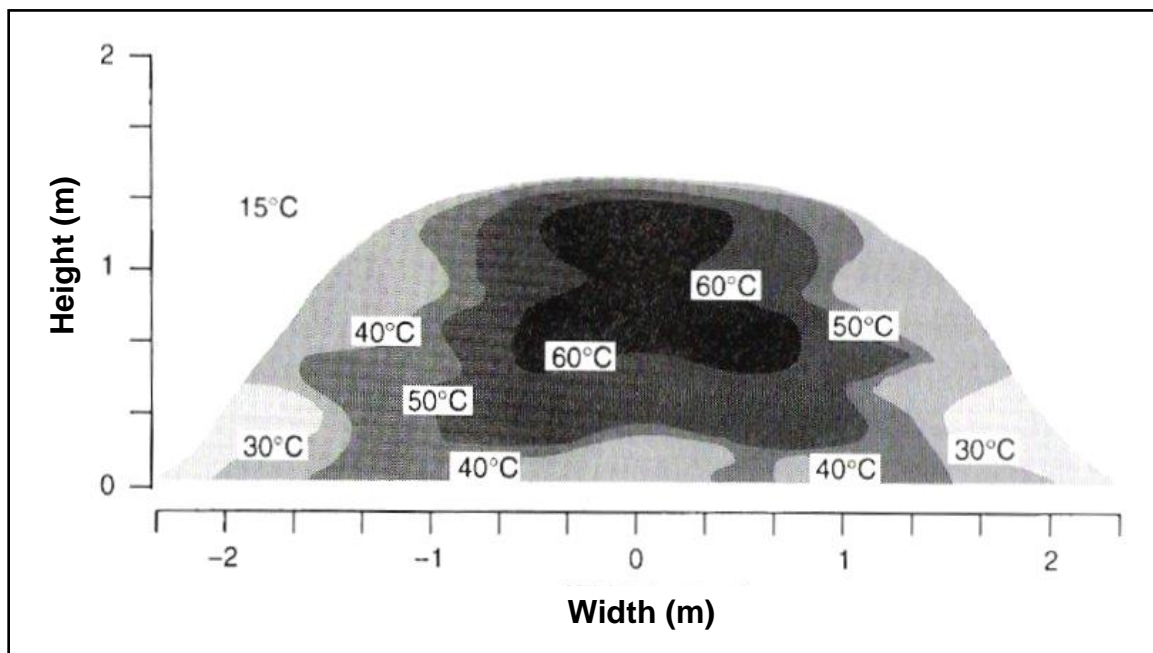
Figure 1.2 Temperature profile of a static compost pile/windrow after 2 - 3 days and during the thermophilic phase (Diaz *et al*., 1993)

When the temperature increases over 40°C, thermophilic bacteria take over and thermophilic fungi also appear. Proteins and the more resistant compounds such as cellulose, hemicellulose and lignin are degraded and partly transformed into humus (McKinley and Vestal, 1985; Crawford, 1983 in Tuomela *et al.*, 2000). Thermophilic bacteria prominent in the composting process are *Bacillus stearothermophilus, Thermomonospora* and *Clostridium thermocellum* as well as *Thermoactinomyces* (the

9

fungi-like bacteria that grow mycelia and produce spores). Important fungi in the thermophilic composting phase are *Geotrichum candidum, Aspergillus fumigatus, Mucor pusillus, Chaetomium thermophile, Thermoascus auranticus* and *Torula thermophila* (Atlas and Bartha, 1998). When the temperature exceeds 60°C, microbial activity decreases dramatically, but after the compost has cooled, mesophilic bacteria and actinomycetes again dominate with some fungi present (McKinley and Vestal, 1985; Crawford, 1983 in Tuomela *et al.*, 2000).

Conducting the composting process in the thermophilic temperature range (generally considered to be above 45°C) is desirable, because it speeds up the chemical and biochemical reactions that drive the process. Cellulose and other more resistant materials are decomposed in the thermophilic (active) phase and it is essential to maintain good aeration to assure even heating of all the material (Brady and Weil, 1999). Maintenance of high temperatures is also the primary mechanism for pathogen inactivation and seed destruction. Regulations of the United States of America Environmental Protection Agency (US EPA) lists composting as a process that significantly reduces pathogens in WWS if the temperature of the sludge is raised to 40°C or higher, remains at this temperature for five days and if the temperature in the compost pile exceeds 55°C for four hours during the five days. Composting will further reduce pathogens if the temperature of the sludge is maintained at 55°C or higher for three days for in-vessel and aerated static pile methods and if the temperature is maintained at 55°C or higher for 15 days or longer with a minimum of five turnings during this period for windrow composting (Electronic Code of Federal Regulations, 2007). Most plant pathogens will be destroyed at 55°C and the critical temperature for destroying weed seeds is approximately 60°C (Cundiff and Mankin, 2003).

Under certain conditions, which include sufficient mass and low heat conductivity of the composting material, self-heating may raise temperatures inside a compost pile to 76-78°C. Temperatures this high are actually inhibitory to biodegradation because maximal thermophilic activity occurs between 52°C and 63°C. Aeration or turning (Figure 1.3) may be adjusted to prevent excessive self-heating and periodic water spraying can also

reduce the temperature (Atlas and Bartha, 1998). Inadequate self-heating can occur when the substrate is not sufficiently balanced in terms of nutrients, water or oxygen, thus retarding microbial growth. Excessive heat loss, especially on the outside of the compost pile, can be overcome by covering the pile with a blanket of finished compost or other suitable material (Atlas and Bartha, 1998).

Figure 1.3 Feedstock of dining hall food scraps being composted in large-scale windrows that are mechanically turned/aerated (Brady and Weil, 1999)

Adequate water (50 - 60% of wet mass) must be present to meet the physiological needs of the micro-organisms, however, excessive water (70% or more) should be avoided as it interferes with aeration and lowers self-heating because of its large heat capacity (Golueke, 1982a).

11

Senn (1971) illustrated the importance of initial feedstock water content during composting of dairy manure in aerated bins. Feedstock loaded at 66% water content only achieved a temperature of 55°C while feedstock loaded at 60% water content quickly rose to above 75°C and remained there for several days. When the wetter feedstock was removed, dry material added and the bin reloaded at 61% water content, the temperature also rapidly increased to above 75°C. They concluded that the higher water content reduced feedstock porosity due to "packing" - impeding air movement through the material.

After some time at peak temperature, there is a gradual temperature decline and the thermophilic phase is followed by a secondary mesophilic phase of several weeks to months of "curing". During this period, the thermophilic populations decline, and the material is re-colonised by mesophiles that survived the thermophilic conditions (Brady and Weil, 1999).

The aerated pile process achieves substantially faster composting rates through improved aeration (Figure 1.4). The process involves suction of air through perforated pipes buried inside the compost pile or reversing the airflow from suction to injection. Thermostats placed inside the pile, control blower operation starting for example, when the temperature exceeds 60°C. The injection of air not only oxygenates the pile, but also cools it sufficiently to avoid self-limiting temperatures. The aerated pile process goes to completion in about three weeks and could be hastened considerably by enriching the input air stream with pure oxygen (Atlas and Bartha, 1998). Suction has the advantage that discharge air can be vented into a small pile of sawdust, wood chips or finished compost, which then serves as an odour filter (Golueke, 1982c).

Composting can be accomplished more rapidly using a bioreactor. This process forms a uniform and stable product, but it also requires a high initial investment. The reactor is maintained at thermophilic temperatures, using the heat produced in the compost process. Composting in the reactor is apparently accomplished in two to four days followed by "curing" for about a month prior to packaging and shipment (Atlas and Bartha, 1998).

Figure 1.4 Sewage sludge and wood chip feedstock being composted by a static (unturned), aerated pile/windrow method. Air is drawn through the windrow via perforated pipes connected to small blowers and expelled through piles of finished compost that absorb odours (Brady and Weil, 1999)

An understanding of the microbial ecology in the compost process can be useful to optimise the process, and standardised microbiological analyses may potentially be used for judging the quality and maturity of compost (Sundh and Ronn, 2002). The enumeration technique ("plate count method") traditionally employed to characterise micro-organisms is highly selective, but recent cultivation independent molecular techniques enable the identification of new organisms, and more importantly, the quantification of shifts in microbial community structure (Cooper, Anderson and Campbell, 2002).

Sundh and Ronn (2002) report that the analysis of signature phospholipid fatty acids is an excellent tool for following the overall microbial succession during composting, and

13

allows a quantitative description of the development of the total microbial biomass, as well as of different groups of eubacteria (including actinomycetes) and eukaryotes.

1.5 Physical and chemical characteristics of substrates and feedstock

In the case of solid wastes, particle size is important and substrates are often shredded to increase the surface to mass ratio in order to facilitate contact between substrate and microbes for effective bio-degradation. Problems regarding size reduction arise from the energy and monetary costs of the operation. Secondly, the mechanical strength of the substrate will determine its structure and porosity with implications for the amount of aeration required, as well as the amount of moisture permissible before negatively impacting on its structure through clumping and/or collapse (Golueke, 1982c).

Composting of municipal and industrial WWS can present unique problems because the organics may still contain 70% or more water. The high water content, lack of porosity, tendency to compact, and the need to dry dewatered sludge during composting require special consideration for (1) structural conditioning of the feedstock to achieve a friable mixture of materials and (2) energy conditioning to close the thermodynamic balance. The advantages are that sludge tends to be relatively homogeneous and free of trash/foreign materials. It also typically contains adequate nutrients to support composting and usually very little pre or post processing is required beyond dewatering (Haug, 1993).

1.6 Physical/structural conditioning of composting feedstock

Structural conditioning and drying of wet substrates such as WWS can be accomplished by adding amendments like wood shavings, straw, peat, cotton gin trash, refuse fractions, yard and agricultural wastes, vermiculite, and a variety of other waste materials. A specific kind of amendment called a bulking agent is an organic or inorganic material of sufficient size to provide structural support and maintain air spaces within the composting matrix by particle-to-particle contact. Wood chips about three to five centimetres in size are the most commonly used bulking agent for sludge composting, although the use of other materials, such as shredded rubber tyres, nut shells, tree trimmings and paper pulp,

14

has been reported (Haug, 1993). Non-degradable or slowly degradable bulking materials are re-used once separated from the finished compost by screening. Availability, material cost and additional handling costs, as well as the suitability of bulking materials in obtaining the desired physical and chemical properties when mixed with the sludge, are primary considerations in their choice. Compost product recycle can be used to reduce the bulk weight and water content of a feedstock and thereby reduce the quantity of supplemental amendments required. It is important to know the characteristics of amendments and bulking agents added to the process (Haug, 1993).

Conditioning of the feedstock is a balancing act between various factors. Physical or structural conditioning aims to eliminate the potential limitations of oxygen and/or water availability to microbial growth during the composting process. Haug (1993) presents considerable evidence that the optimum moisture content for a particular substrate is related to maintenance of a certain minimum free air space (air-filled porosity). A minimum free air space of ~30% should be maintained to achieve about 95% of the maximum oxygen consumption rate.

Using amendments and/or compost recycle to adjust feedstock (mixture) solids content to the range of 35 to 45% on a wet mass basis (corresponding to water content of 65 to 55%) is common practice with wet substrates. When a bulking agent is considered for use, the capacity for water absorption by the bulking agent and the extent to which the sludge releases (desorbs) water, have to be taken into account. The lesser of these will determine how much water the bulking agent will absorb. The absorbed water will not influence the air filled porosity of the bulking agent matrix, and together with the water content of the sludge and the total porosity of the bulking agent matrix, the mixing ratio of sludge to bulking agent can be determined to obtain a minimum free air space of ~30%. The volume of bulking agent should theoretically not increase after mixing with sludge because the sludge will occupy only about 70% of the total porosity of the bulking agent matrix (Haug, 1993). Evaporative drying during composting necessitates water addition to avoid dehydration of substrates to the point where the composting process is negatively impacted.

1.7 Energy conditioning of feedstock

The energy released from organic decomposition drives the composting process. It causes temperature elevation and facilitates drying of the material. Energy conditioning of the feedstock to ensure that sufficient energy is available is critical when composting wet substrates because of the large energy burden placed on the system to support evaporative drying. To judge the need for energy conditioning without conducting mass and energy balances the following rules of thumb can be employed (Haug, 1993):

- The water ratio (W) is defined as the ratio of water to BVS. Defining W on the basis of water content is a rational approach because evaporation represents most of the total energy demand under conditions expected with composting and drying of wet substrates. To judge the thermodynamic characteristics of the compost process, a W ratio of about 8 to 10 g $H_2O.g^{-1}$ BVS can be used as long as water in the substrate is the major water input. Sufficient energy should be available for temperature elevation and water evaporation if $W < 8$ $g.g^{-1}$. The substrate alone may not provide sufficient energy if $W > 10$ $g.g^{-1}$ and lower process temperatures or less drying can be expected.

- The energy ratio (E) is defined as the ratio of heat released by BVS to the water content. The E ratio removes the limiting assumption (used with the W ratio) that all substrate organics have the same heat value by including the heat content (H) in the analysis. An E ratio of about 2933 $J.g^{-1}$ H_2O can be used as a measure of the energy content (and therefore the compostability) of the substrate. Sufficient energy should be available for both composting and drying if $E > 2933$ $J.g^{-1}$ H_2O and drying may have to be reduced to maintain process temperatures if $E < 2514$ $J.g^{-1}$ H_2O.

Estimating heats of reaction for organic wastes is often difficult from standard enthalpy values because they comprise a mixture of organics of unknown composition. Open and bomb calorimetric techniques can be used experimentally to determine the heats of decomposition for such unknown materials. However, a number of empirical equations are available which yield reasonably consistent results of sufficient accuracy for most analyses of sludge composting systems (Haug, 1993). A general rule of thumb for sewage

16

sludge presented by Vesilind (1979) and others is to expect about 23.2 kJ.g^{-1} dry volatile solids. This is, however, based on a typical sludge composition of proteins, carbohydrates and fats that may vary - particularly with large inputs of industrial waste and other pre-composting stabilisation techniques.

Substrate biodegradability can be estimated by Chandler's predictive model (Chandler *et al.*, 1980). Using data from the anaerobic fermentation of a wide range of lignin protected/associated cellulosic (ligno-cellulosic) material, a linear relationship between the biodegradability of organic substrates and their lignin content was obtained:

$$B = 0.830 - 0.028X$$

Where B is the biodegradable fraction of volatile solids and X is the lignin content, as % of volatile solids (Haug, 1993; Tuomela *et al.*, 2000).

1.8   Chemical conditioning of feedstock

Chemical conditioning aims to eliminate the potential limitations of nutrient availability and/or pH, to microbial growth. Carbon is important in microbial cell synthesis to build up protoplasm, oils, fats and carbohydrates as well as in microbial metabolism where it is oxidised to yield energy and CO_2. Nitrogen, a constituent of protein, is essential to cell survival and reproduction (Golueke, 1982b). Other important nutrients are phosphorous (found in energy-rich compounds), calcium and the micronutrients cobalt, zinc, chromium and molybdenum, which play an important role in enzyme systems and intra- and extra-cellular transport (Golueke, 1982b). Materials often added to compost to improve nutrient balance and content are mineral fertilisers, wood ashes, bone meal or phosphate rock powder and seaweed (Brady and Weil, 1999).

Nitrogen is the inorganic nutrient required in the highest concentrations and has received the most attention in composting systems. The composting industry generally uses the carbon to nitrogen ratio (C:N) as a measure of nitrogen availability for the process of biological degradation. Sound theoretical arguments (living organisms use about thirty

17

parts carbon for each part nitrogen) supported by actual field experiments sets the C:N requirement for rapid composting at around 25:1 to 30:1. Above this range, composting time usually increase with increasing C:N ratio due to growth limitations imposed by a lack of nitrogen. Below this range nitrogen is present in excess and no rate limitation is imposed but some nitrogen is likely to be lost as volatilised ammonia or nitrous oxide in the exhaust gases. This may also result in odour problems (Cundiff and Mankin, 2003; Atlas and Bartha, 1998).

Use of the C:N ratio as stated above, assumes that both the carbon and nitrogen sources are relatively biodegradable. Seeing that most of the nitrogen in naturally occurring substrates is present in relatively degradable protein molecules the assumption generally holds true for nitrogen. It is, however, well known that carbonaceous compounds like lignin and ligno-cellulose are not readily decomposed by micro-organisms and that they are usually present at the end of typical composting times (Haug, 1993). Carbonaceous compounds resistant to microbial degradation imply a delayed or reduced nitrogen need and thus a higher C:N ratio is possible without retarding microbial growth (Golueke, 1982b).

Haug (1993) proposed the use of the Chandler *et al.*, (1980) equation to adjust the C/N ratios of materials for compost mixture calculations to biodegradable carbon instead of total carbon; especially where plant materials and food waste is a component of the composting feedstock. The linear relationship given by Chandler *et al.* (1980) is simple and appears to provide reasonable accuracy for materials of relatively low lignin content. With a large amount of lignin present, some of the lignin would be overlapping other lignin molecules rather than cellulose, so the incremental effect will be smaller (Conrad *et al.*, 1984). While virtually no lignin is degraded in anaerobic environments (Essington, 2004), studies have shown that significant lignin degradation is possible in aerobic (soil, compost) environments and it can be expected that the restriction on substrate biodegradability in these environments will be less.

18

Some researchers have studied the effect of composting temperature on lignin degradation. In a laboratory incubation study Horwarth and Elliott (1996) composted ryegrass straw for 45 days and determined lignin degradation (Klason method) to be 7% when the temperature remained at 25°C for the whole period, and 27% when the temperature was at 50°C for 30 days and at 25°C for 15 days. At both temperature profiles, the elemental ratio of the residual lignin changed, and they estimated that only 6% of the remaining lignin was unaltered after composting (Tuomela *et al.*, 2000).

The data of Waksman, Cordon and Hulpoi (1939) is presented in Figure 1.5. The highest lignin degradation in horse manure and straw compost occurred at 50°C (43%) and degradation was somewhat lower at 28°C (26%) and 65°C (33%) during 50 days of incubation. It was concluded that no biodegradation of lignin occurred at 75°C, but that some 12% of the lignin was solubilised due to the high temperature and alkaline reaction of the compost (Tuomela *et al.*, 2000).
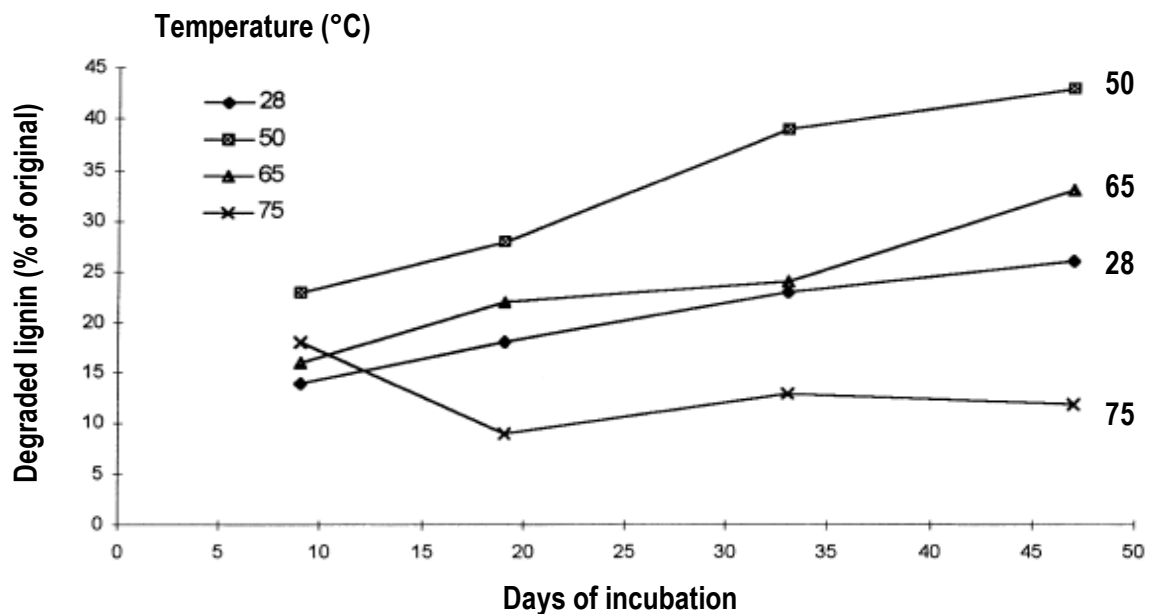
Figure 1.5 Lignin degradation in horse manure and straw feedstock (original lignin content 20.5%), data derived from Waksman *et al.*, (1939) (Tuomela *et al.*, 2000)

19

Tomati *et al.* (1995) reported 70% lignin degradation in olive mill waste and wheat straw compost during 35 days at 50°C. Negligible degradation during the subsequent 67-day maturation period at mesophilic conditions was observed.

These and other studies clearly show that significant lignin degradation and alteration can occur in composting environments. The environmental factors that determine microbial growth such as water content, oxygen and nutrient/substrate availability and pH, are usually within acceptable limits in a well-engineered composting system. Therefore, temperature, original lignin content and particle size or thickness of the material (Robinson *et al.*, 1994; Franzluebbers, Arshad and Ripmeester, 1996 in Tuomela *et al.*, 2000), seems to be important factors controlling lignin degradation during composting. Furthermore, a host of empirical data produced over an extensive period of research and a large variety of substrates with varying degrees of degradability, seems to support a total C:N ratio of 25:1 to 30:1 (Haug, 1993).

Excessive amounts of micronutrients or other heavy metals can be toxic to micro-organisms and organic acids can unfavourably alter the pH, thereby limiting process dynamics during composting. Compost generally has the rather unique ability to buffer extremes of pH and pH adjustment of the starting substrates is usually not required. This ability stems from the fact that both carbon dioxide (a weak acid) and ammonia (a weak base) are released as a result of organic decomposition (Haug, 1993).

1.9   Monitoring and management of the composting process

From the discussion of composting process principles and conditions so far, the key process variables that require monitoring and management have been identified and are presented in Table 1.2 in relation to the processing steps during composting at which they are of importance.

For detail discussions on the importance of sampling, different sampling strategies, sample handling and preparation as well as analytical techniques for the monitoring of

20

these key process variables, refer to Test Methods for the Examination of Composting and Compost (Thompson *et al.*, 2002).

Table 1.2 Management of key process variables, re-produced from Thompson *et al.*, 2002

KEY PROCESS VARIABLES	Feedstock	PROCESSING STEPS						
	Feedstock collected and delivered	Step 1: Feedstock recovery	Step 2: Feedstock preparation	Step 3: Composting	Step 4: Odour treatment	Step 5: Compost curing	Step 6: Compost screening and refining	Step 7: Compost storing and packaging
Pile porosity	✓	✓	✓	✓	✓	✓	✓	✓
Feedstock nutrient balance	✓	✓	✓					
Pile oxygen %			✓	✓	✓	✓		✓
Pile water %			✓	✓	✓	✓	✓	✓
Pile temperature				✓		✓		
Retention time			✓	✓	✓	✓		✓

1.10 Summary

The term "composting" refers to the aerobic biological decomposition of organic materials which requires oxygen and produces carbon dioxide, water and heat. Both mesophilic and thermophilic micro-organisms contribute to composting and the process is most rapid when conditions that encourage their growth, are established and maintained. Such conditions require a mixture of materials (feedstock) that provide the nutrients needed for microbial activity and growth, including a balanced supply of available carbon and nitrogen, enough oxygen to keep the process aerobic, and enough water to permit biological activity without hindering aeration.

Composting occurs to various degrees over a wide range of conditions. Conducting the composting process in the thermophilic temperature range (generally considered to be above 45°C) is desirable, since it speeds up the chemical and biochemical reactions that drive the process. Maintenance of high temperatures is also the primary mechanism for pathogen inactivation and seed destruction. After some time at peak temperature, there is a gradual temperature decline and the active (thermophilic) phase is followed by a secondary mesophilic phase of several weeks to months of "curing".

Composting of municipal and industrial WWS can present unique problems because the organics may still contain 70% or more water. The high water content, lack of porosity, tendency to compact, and the need to dry dewatered sludge during composting require special consideration for (1) structural conditioning of the feedstock to achieve a friable mixture of materials and (2) energy conditioning to close the thermodynamic balance.

Structural conditioning and drying of wet substrates such as WWS can be accomplished by adding amendments like wood shavings, straw, peat, cotton gin trash, refuse fractions, yard and agricultural wastes, vermiculite, and a variety of other waste materials. A specific kind of amendment called a bulking agent is an organic or inorganic material of sufficient size to provide structural support and maintain air spaces within the composting matrix by particle-to-particle contact. A minimum free air space of ~30% should be maintained to achieve about 95% of the maximum oxygen consumption rate. Using amendments and/or compost recycle to adjust feedstock solids content to the range of 35 to 45% on a wet mass basis (corresponding to a water content of 65 to 55%) is the common practice with wet substrates.

The energy released from organic decomposition drives the compost process, causes temperature elevation and facilitates drying of the material. Energy conditioning of the feedstock to ensure that sufficient energy is available is critical when composting wet substrates because of the large energy burden placed on the system to support evaporative drying.

Chemical conditioning aims to eliminate the potential limitations of nutrient availability and/or pH, to microbial growth. The composting industry generally uses the carbon to nitrogen ratio (C:N) as a measure of nitrogen availability for the process of biological degradation and sets the C:N requirement for rapid composting at around 25:1 to 30:1. Excessive amounts of micronutrients or other heavy metals can be toxic to micro-organisms and organic acids can unfavourably alter the pH, thereby limiting process dynamics during composting. Compost generally has the rather unique ability to buffer extremes of pH and pH adjustment of the starting substrates is usually not required.

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25

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Chapter 2: Compost safety and quality – a review

2.1 Introduction

A precise definition of compost quality is often controversial. It can be described as *the summation of the numerous characteristics of compost that are recognised as playing a role in sustaining soil quality and contributing to a plant's good health and yield* (Balis, Tassiopoulou and Lasaridi, 2002). Haug's definition of compost reveals some more quality characteristics when he writes: *Compost is an organic material that has been stabilised to a humus-like product, that is free of viable human and plant pathogens and plant seeds, that does not attract insects or vectors, that can be handled and stored without nuisance, and that is beneficial to the growth of plants* (Haug, 1993).

Compost can be highly variable in its physical, chemical and microbiological make-up, with definite implications for its quality and functionality in application as well as its safety to plants, animals, humans, and the soil and aquatic environment. These characteristics of compost are intrinsically linked to initial substrate characteristics, management of the composting process and environmental conditions during composting, as well as the maturity of the product (Itavaara *et al*., 2002).

The aim of this literature study is to present a framework for compost quality in terms of South African quality standards with specific reference to the mechanisms, risks and advantages involved in pathogen inactivation (sanitation), maturity (stabilisation) and chemical / biochemical and organic/ biological quality characteristics of compost.

2.2 Compost quality standards

Quantitative quality criteria are seldom agreed upon and are usually specified in terms of minimum admissible levels of required substances or maximum tolerable limits for unwanted ones. Ultimately the intended compost use will determine which of the various quality criteria will be relevant (Balis *et al*., 2002). Apart from soil application, compost can also be used as mulch, top dressing and as a component of container growth media (Haug, 1993).

Product standards are necessary to protect public and environmental health and to assure a measure of commercial acceptability. Primary pathogens exist in the materials to be composted and secondary pathogens can grow during the composting process. Heavy metals and trace organics contained in compost can enter the food chain through plants. Several characteristics of compost are important to commercial acceptance but do not affect human health or the environment. These include colour, particle size, the presence of weed seeds and other foreign or inert material, organic matter content and its stability (compost maturity), carbon to nitrogen ratio, salinity, pH, water content, and water holding capacity (Haug, 1993).

The South African statutory requirements for the registration and marketing of compost are set out in Part II of Annexure A of Government Notice R.250: Regulations regarding fertilisers (Fertilisers, Farm Feeds, Agricultural Remedies and Stock Remedies Act, Act 36 of 1947) that deals with the requirements for registration of organic fertilisers. These are as follows:

- The ash content must not exceed 400 g.kg^{-1} (40%) on a dry mass basis;
- The water content must not exceed 40 g.kg^{-1} (40%) on a wet mass basis;
- It must not contain any visibly un-decomposed organic or other foreign material;
- At least 80% of the seeds (type not specified) that are planted under controlled conditions germinate normally and exhibit normal growth when planted in a growth medium as prescribed by the manufacturer/registration holder);
- If it is sold in containers, it must be fine enough for 100% thereof to pass through a 12 mm standard sieve;
- If it is sold as a household and garden fertiliser as intended in Regulation 1 of the regulations relating to fertilisers, the 12mm size regulation shall apply whether it is sold in containers or in bulk.

Act 36 of 1947 further stipulates that compost that consists of sewage sludge can be registered and sold if it is a "type D sludge" and if it meets the requirements for total metal and inorganic content as described and listed in Table 12 of the Act. Sewage sludge

compost must also meet the requirement of 80% seed germination (as stated above) and no macro- or micro-elements may be added to the sludge without the written approval from the Registrar (Fertilisers, Farm Feeds, Agricultural Remedies and Stock Remedies Act 36, 1947).

The more recent "Guidelines for the utilisation and disposal of wastewater sludge" (Snyman and Herselman, 2006) stipulate a three-way classification system for WWS (and sludge compost) namely 1) microbiological class, 2) stability class and 3) pollutant class.

The beneficial use of sludge is encouraged throughout the guidelines to ensure sustainable sludge management and part B3, Volume 5 deals with fertiliser products containing sludge, including sludge compost, that are distributed to the general public for unrestricted use. Regulating the quality of the final product is of paramount importance and the product must be sampled and analysed before distribution to the public and must comply with **Class A1a** according to the classification system of the guidelines (verbatim as follows):

- **Microbiological class A** requires a value for Faecal coliform < 1000 CFU.g_{dry}^{-1} at a 90% compliance level and a value for Helminth ova (viable ova. g_{dry}^{-1}) < 0.25 (or 1 ova/4g_{dry}) at a 90% compliance level and in both the 10% samples that exceed these target values may not exceed maximum permissible values of 10 000 and 1.

- **Stability class 1** requires that the product must always comply with one of seven "vector attraction reduction options" namely:
  Option 1: Reduce the mass of volatile solids by a minimum of 38%;
  Option 2: Demonstrate vector attraction reduction with additional anaerobic digestion in a bench-scale unit;
  Option 3: Demonstrate vector attraction reduction with additional aerobic digestion in a bench-scale unit;
  Option 4: Meet a specific oxygen uptake rate for aerobically treated sludge;

30

Option 5: Use aerobic processes at a temperature greater 40°C (average temperatures 45 °C) for 14 days or longer (e.g. during sludge composting);

Option 6: Add alkaline material to raise the pH under specific conditions;

Option 7: Reduce moisture content of sludge that does not contain un-stabilised solids (from treatment processes other than primary treatment) to at least 75% solids.

- **Pollutant class a** requires values for eight *Aqua regia* extractable metals (mg.kg^{-1}) below: Arsenic (40), Cadmium (40), Chromium (1 200), Copper (1 500), Lead (300), Mercury (15), Nickel (420) and Zinc (2 800).

The guidelines further recommend that compost batches are sampled and analysed for its Microbial classification before it enters the curing phase as the high temperatures during the active phase should have deactivated all the helminth ova by this time. Compost produced in a properly designed and operated plant should readily comply with at least Options 2 and 5 to qualify as Stability class 1. Batches that don't comply with the pollutant class limits could be sufficiently blended with compost that does comply. Sampling protocols, test methods, vector attraction reduction options etc. are stipulated and discussed in the guidelines (Snyman and Herselman, 2006).

Since the decomposed bulking agent adds relatively little to the mass of the compost, the pollutant concentration in the compost stays essentially the same as in the feedstock sludge and therefore, it is unlikely that **Class A1a** compost will be produced if the sludge used does not comply with **Pollutant class a** (Snyman and Herselman, 2006).

2.3   Pathogens

One of the many benefits to be derived from composting is the transformation of unsafe wastes into a harmless and useful product. Four factors regarding pathogen and parasite destruction during composting are considered important: high temperature, interspecific competition, antibiosis and time (i.e. length of exposure). High temperature is the most effective in pathogen-kill and it is generally accepted that damp heat is more lethal than dry heat (Golueke, 1982a). The problem with windrow composting is the occurrence of

31

temperature zones, grading from high temperatures in the core of the pile to ambient temperatures at the outer zones. Cooper and Golueke (1982) reported that in large-scale windrows with temperatures of 65°C in the interiors, an estimated 25% of the total mass was at temperatures below 50°C. Their studies also confirmed that a lack of moisture retarded pathogen kill even if the temperature was sufficiently high.

Baccella *et al*. (2002), report results that indicate the possibility to obtain good sheep litter sanitation by adding actinobacterial strains, isolated from soil, at the beginning of the composting process. Laboratory tests showed that some selected strains are able to inhibit *Escherichia coli* and several pathogen clostridial strains native to the litter.

The disease botulism in man and animals is caused by neurotoxins produced by *Clostridium botulinum*. The classical form of this normally fatal disease is the inhibition of neurotransmitter release at the neuromuscular synapses, which lead to paralysis of muscles. Bohnel, Briese and Gessler (2002), tested 143 samples of compost and commercial potting soils containing compost. In 66 of the samples *C. botulinum* was detected. *C. botulinum* is a soil bacterium that may proliferate directly in soil and may survive for decades by spore formation. It multiplies in the intestines of animals and humans and is spread by faecal excretion (Bohnel *et al*., 2002).

From their investigation into the suitability of composting to recycle horticultural waste and at the same time suppress the infective capacity of several harmful plant pathogens, Suarez-Estrella *et al*. (2002) reported results that showed an effective suppression of viral infectivity of melon necrotic spot virus during the initial 14 days of composting. Short interval sampling demonstrated suppression of tomato spotted wilt virus after 60 hours of composting, whereas pepper mild mottle virus was the most resistant pathogenic virus, remaining viable up to eight to nine weeks. In all cases, viral inactivation was achieved during the active phase of composting (before the maturation or curing phase), suggesting the need to perform waste composting processes in which high temperatures are maintained over several weeks.

From Chapter 1: Regulations of the US EPA list composting as a process that significantly reduces pathogens in WWS if the temperature of the sludge is raised to 40°C or higher and remains there for five days and if the temperature in the compost pile exceeds 55°C for four hours during the five days. Composting will further reduce pathogens if the temperature of the sludge is maintained at 55°C or higher for three days for in-vessel and aerated static pile methods and if the temperature is maintained at 55°C or higher for 15 days or longer with a minimum of five turnings during this period for windrow composting (e-CFR, 2007). Most plant pathogens will be destroyed at 55°C and the critical temperature for destroying weed seeds is approximately 60°C (Cundiff and Mankin, 2003).

It seems that, with the simultaneous action of all four factors considered here, sufficient pathogen-kill could be expected if good aerobic, thermophilic composting principles are practised. In some cases where a particular unwanted pathogen poses a problem by surviving the composting process, the possibility exists to inoculate the compost pile with a microorganism known to produce antibiotics, or to be a strong competitor.

2.4 Compost maturity

Compost quality is closely related to its stability and maturity as it affects a number of its agricultural properties, like phytotoxicity, nutrient availability, suppressiveness against soil-borne pathogens, etc. (Balis *et al*., 2002). The active phase of composting is characterised by partial mineralisation and stabilisation of the organic matter. The curing phase is characterised by the conversion of part of the stabilised organic matter into humic substances and it is this phase that affects compost maturity most (Cunha Queda *et al.*, 2002).

Humic substances comprise an extraordinarily complex, amorphous mixture of highly heterogeneous, chemically reactive yet refractory molecules, produced during early diagenesis in the decay of biomatter, and formed ubiquitously in the environment via processes involving chemical reaction of species randomly chosen from a pool of diverse

33

molecules and through random chemical alteration of precursor molecules (MacCarthy, 2001).

Indicators of compost stability and maturity are important tools to define the end point of composting and to evaluate compost quality. Different methods and parameters have been proposed. They range from chemical tests, microbiological assays and higher plant bioassays, to humified organic matter analyses (Balis *et al.*, 2002). Some of these methods like the *self-heating test* and parameters like *oxygen consumption rate*, aim to detect residual microbial activity, while others like the *cress (Lepidium sativum) seed germination index* is a measure of phytotoxicity, which has been widely used and considered as a reliable indirect quantification of compost maturity (Cunha Queda *et al.*, 2002).

Brinton and Evans (2002) studied the effect of compost of varying maturity on plant performance. Their data suggest that a number of important, interrelated factors played a role in the poor plant and root development in immature compost. The causal mechanism was most likely elevated CO_2 evolution and volatile fatty acid production, elevated ammonia levels, along with oxygen deprivation and hydrogen sulphide production in containers during growth.

2.5   Chemical/biochemical and organic/biological quality characteristics

Compost contains valuable nutrients including nitrogen, phosphorus, and a variety of essential trace elements and its nutrient content is related to the quality of the original organic substrate. Although compost generally contains insufficient nutrients to be classified as fertilisers, the organically bound nutrients are slowly released throughout the growing season with obvious environmental and nutritional advantages. After compost incorporation in soil (1) certain organic substrates not degraded during the composting process can readily mineralise (2) C and N cycling can be affected by compost feedstock, processing conditions, and time, and (3) de-nitrification after compost incorporation in soil can limit N availability from compost (Cambardella, Richard and Russell, 2003). A phenomenon termed nitrogen "draw-down" or immobilisation can temporarily limit N

34

availability from compost when it contains insufficient N in relation to its degradable carbon content (Brady and Weil, 1999).

Chemical properties that can be considered/analysed for compost quality assessment includes its content of organic carbon, inorganic carbon, nitrogen, phosphorus, potassium, secondary and micro-nutrients, heavy metals and hazardous elements, cation exchange capacity, electrical conductivity, pH and salinity or soluble salt content (Thompson *et al*., 2002). They also give detail descriptions of digestion techniques for atomic absorption spectrometry and inductively coupled plasma analysis.

The more complex organic polymers like lignin are considered partially responsible for the physical properties of the final compost (Cundiff and Mankin, 2003). In plant residue decomposition studies, lignin is considered to be the most important component determining the rate of decomposition. It is suggested that the lignin-to-nitrogen ratio predicts the early stage of decomposition, but the lignin content alone or the ligno-cellulose index [lignin:(lignin + holocellulose)] predicts the longer-term decomposition rate (Palm and Rowland, 1997).

Other resistant complex organic polymers found in compost are humic substances. Unlike lignin, humic substances are synthesis products from the microbial decomposition of organic materials and its content is considered to markedly increase during the composting process (Brady and Weil, 1999). Humic substances are partitioned into three main fractions namely humic acid, fulvic acid and humin, based on their differential aqueous solubility in acidic and alkaline solutions and according to Essington (2004), not by their innate structural or chemical characteristics.

Tuomela *et al*. (2000), report that immature compost generally contains high levels of fulvic acids and low levels of humic acids. The humic acid fraction increases as decomposition proceeds while the fulvic acid fraction either decreases or remains unchanged. Aliphatic material is lost by mineralization while enrichment in aromatic structures occurs (Garcia et al., 1992).

35

The ratios between humic acid carbon (HAC) and fulvic acid carbon (FAC), as well as a number of other ratios involving HAC, FAC, total organic carbon (TOC) and alkali extractable carbon (EXC) have been used to evaluate compost maturity and the degree of humification in composting substrates at various stages of the process (Sanchez-Monedero *et al.*, 1999; Govi *et al.*, 1993).

Organic and biological properties that can be considered/analysed for compost quality and safety assessments includes biodegradable volatile solids, colour, enzyme activity and analysis, biological assays, odour, organic matter, respirometry, viable weed seeds, volatile fatty acids as well as synthetic organic compounds like chlorinated herbicides, dioxin/furans, organochlorine pesticides, organophosphorus pesticides, polychlorinated biphenyls, semi-volatile organic compounds and volatile organic compounds (Thompson *et al*., 2002).

2.6   Summary

Compost can be highly variable in its physical, chemical and microbiological make-up, with definite implications for its quality and functionality in application as well as its safety to plants, animals, humans, and the soil and aquatic environment. These characteristics of compost are intrinsically linked to initial substrate characteristics, management of the composting process and environmental conditions during composting, as well as the maturity of the product. Product standards are necessary to protect public and environmental health and to assure a measure of commercial acceptability.

The South African statutory requirements for the registration and marketing of compost are set out in the *Fertilisers, Farm Feeds, Agricultural Remedies and Stock Remedies Act (Act 36 of 1947)* with emphasis on product quality and in the "Guidelines for the utilisation and disposal of wastewater sludge" (2006) that stipulate a three-way classification system for WWS (and sludge compost) with emphasis on product safety.

Four factors regarding pathogen and parasite destruction during composting are considered important: high temperature, interspecific competition, antibiosis and time

(i.e. length of exposure). It seems that sufficient pathogen-kill could be expected if good aerobic, thermophilic composting principles are practised.

Compost quality is closely related to its stability and maturity as it affects a number of its agricultural properties, like phytotoxicity, nutrient availability, suppressiveness against soil-borne pathogens etc. Indicators of compost stability and maturity are important tools to define the end point of composting and to evaluate compost quality. Methods and parameters range from chemical tests, microbiological assays and higher plant bioassays, to humified organic matter analyses. Methods like the *self-heating test* and parameters like *oxygen consumption rate*, aim to detect residual microbial activity, while others like the *cress (Lepidium sativum) seed germination index* is a measure of phytotoxicity, which has been widely used and considered as a reliable indirect quantification of compost maturity.

Compost contains valuable nutrients and its nutrient content is related to the quality of the original organic substrate. Chemical properties that can be considered for compost quality and safety assessment includes its content of organic carbon, inorganic carbon, nitrogen, phosphorus, potassium, secondary and micro-nutrients, heavy metals and hazardous elements, cation exchange capacity, electrical conductivity, pH and salinity.

Organic and biological properties that can be considered for compost quality and safety assessments includes biodegradable volatile solids, colour, enzyme activity and analysis, biological assays, odour, organic matter, respirometry, viable weed seeds, volatile fatty acids as well as synthetic organic compounds like chlorinated herbicides, dioxin/furans, organochlorine pesticides, organophosphorus pesticides, polychlorinated biphenyls, semi-volatile organic compounds and volatile organic compounds.

The more complex and resistant organic polymers like lignin and humic substances are considered partially responsible for the physical properties of compost (water holding capacity and cation exchange capacity) and play an important role in the decomposition rates of composts.

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Chapter 3: Experimental composting of feedstock consisting of three types of wastewater sludge and Eucalyptus sawdust

3.1 Introduction

The ranges of chemical and physical conditions required for the rapid and successful composting of most organic materials, as well as certain measurable parameters indicative of a successful composting process, are well researched and defined (Chapter 1).

This study proposes that that the compostability of different types of WWS and eucalyptus sawdust bulking agent can be evaluated if a physically and chemically conditioned feedstock is formulated and the composting process conditions appropriately managed through turning and watering while monitoring and evaluating certain composting conditions and feedstock characteristics during composting.

Against this background, a small scale composting experiment was designed to answer the question whether selected WWS from different geographical and process origins could be successfully composted. The study objectives are to:

- Test the application of established composting principles and requirements for the successful composting of three types of WWS in conditions approximating a practical composting situation.
- Elucidate and apply the different requirements for feedstock conditioning and process management for successful composting.
- Evaluate and compare the performance of three types of WWS, during composting with Eucalyptus sawdust as bulking agent.
- Investigate and apply methods and parameters for composting process monitoring.
- Identify useful parameters to predict the suitability of materials for composting.

3.2   Materials and methods

3.2.1 Preliminary composting trial

A preliminary phase composting trial was established using a single feedstock consisting of dewatered (belt press), activated sludge from a municipal water treatment plant (ERWAT Olifantsfontein) and Eucalyptus sawdust as bulking agent. The sawdust was sourced at an industrial site in Olifantsfontein where struts were cut and prepared for use in the mining industry, producing piles of sawdust as a waste product. Due to its proximity to the experimental site and its homogeneous nature it was the most suitable bulking agent that could be sourced from a waste stream in the same geographic area where the chosen WWS originate.

The sludge had an estimated gravimetric water content of 82% (wet mass basis, 18% solids, from discussions with the treatment plant manager) and an assumed C:N ratio of 5.61 (total C of 29.9%, total N of 5.33% on a dry mass basis, determined by dry combustion with a Carlo Elba instrument on a sample from the same source previously collected, one repetition only). The sawdust had a measured water content of 39.2 ± 0.865%, a C:N ratio of 67.0 (total C of 40.9% and total N of 0.61% on a dry mass basis, determined by dry combustion with a Carlo Elba instrument, one repetition only), pH of 4.78 ± 0.165 and EC of 19.0 ± 0.00 $mS.m^{-1}$. It was calculated that a feedstock of sludge and sawdust in a 1:1 ratio (wet mass) would have a water content of 60.6% and a C:N ratio of 22.8 (refer to Appendix A for calculation detail). These were considered to be within acceptable limits for composting. The actual initial water content of the feedstock after mixing was measured to be 59.7% (one repetition only).

The trial consisted of compost piles of different sizes namely 1 m^3, 2 m^3, 3 m^3 and 4 m^3 (which was the largest size that could be accommodated within the facility and handling constraints). Paper pulp (the unusable short fibre by-product from paper recycling) was another possible bulking agent from a waste stream but its source was located much further away and it was known to contain relatively high levels of certain heavy metals (from discussions with and data supplied by Nampak). It had a measured water content of

43

59.7 ± 0.094% (probably too high for use as a bulking agent for dewatered WWS on its own), a C:N ratio of 80.2 (total C of 27.0% and total N of 0.3% on a dry mass basis, determined by dry combustion with a Carlo Elba instrument, one repetition only), pH of 6.90 ± 0.100 and EC of 69.67 ± 5.03 mS.m^{-1}.

To test its effect on pH and temperature development, 0.33 m^3 paper pulp (10% by volume) was added to a 3 m^3 pile. Composting piles were established on a roofed concrete floor at the University of Pretoria Hatfield Experimental Farm. Piles were mixed, turned and watered manually and pile temperature recorded.

The aim with the trial was to test the logistics of raw material measurement, feedstock mixing, pile turning and watering, temperature monitoring and most importantly, to establish the optimal experimental pile size for composting a WWS-sawdust feedstock within the constraints of available space and manual handling and mixing/turning methods, yet large enough not to impact negatively on temperature development (Figure 3.1).

Figure 3.1 The preliminary composting trial of thickened activated sludge from a municipal waste water treatment plant (ERWAT Olifantsfontein) and Eucalyptus sawdust as bulking agent in a 1:1 ratio (wet mass). Left: piles of different sizes (1 m^3, 2 m^3, 3 m^3 and 4 m^3) and; Right: evidence of good heat production during manual pile turning of the 3 m^3 pile with paper pulp added

The temperature profile of different pile sizes (Figure 3.2) shows that, between piles consisting of the same feedstock, pile temperature increased with pile size. There was a large difference (11°C) between the average core temperature of the 1 m^3 pile (43°C) and the 4 m^3 pile (54°C) with the 2 m^3 and 3 m^3 piles neatly between them (48°C for both). The core temperature of all piles was seldom constant and fluctuated in response to changes in ambient temperature and pile turning which occurred on the 5th day (with water addition) and again on the 18th day of composting.

This relationship can be ascribed to an exponential decrease in the surface-to-mass or surface-to-volume ratio with increasing pile size (Figure 3.3). The decrease is large for small piles but quickly becomes relatively small for piles of 4 m^3 and larger (purely from visual interpretation). It can therefore be expected that other mechanisms than pile size will play the dominant role in the temperature profiles of piles larger than 4 m^3.

Apart from its effect on heat transfer from the pile, the pile mass/volume also affected the mass transfer of water through evaporation from the pile. Piles were turned and individually watered according to their look and feel on day 5. When piles were turned again on day 18, the water content was higher in the 4 m^3 pile (49.72 ± 1.27%) than in the 3 m^3 pile (42.85 ± 1.10%) and the 3 m^3 pile containing paper pulp (46.02 ± 5.02%).

The 3 m^3 pile to which 0.33 m^3 of paper pulp was added showed impressive heat production and had a mean core temperature of 58°C. The paper pulp contained an estimated 45% $CaCO_3$ (from discussions with Nampak representative) and the measured pH after 27 days of composting was 6.26 ± 0.038 for this pile, as opposed to pH 5.46 ± 0.113 for the 3 m^3 pile without paper pulp and pH 5.61 ± 0.015 for the 4 m^3 pile (also without paper pulp). Possible explanations for the enhanced heat production observed in the pile to which paper pulp was added include a more favourable pH to microbial growth and activity, a source of easily available Ca (Wong and Fang, 2000) and C stimulating microbial growth or additional water that was probably held at a less negative matric potential (i.e. higher energy state) and thus more available to micro-organisms and for re-distribution in the pile.

45
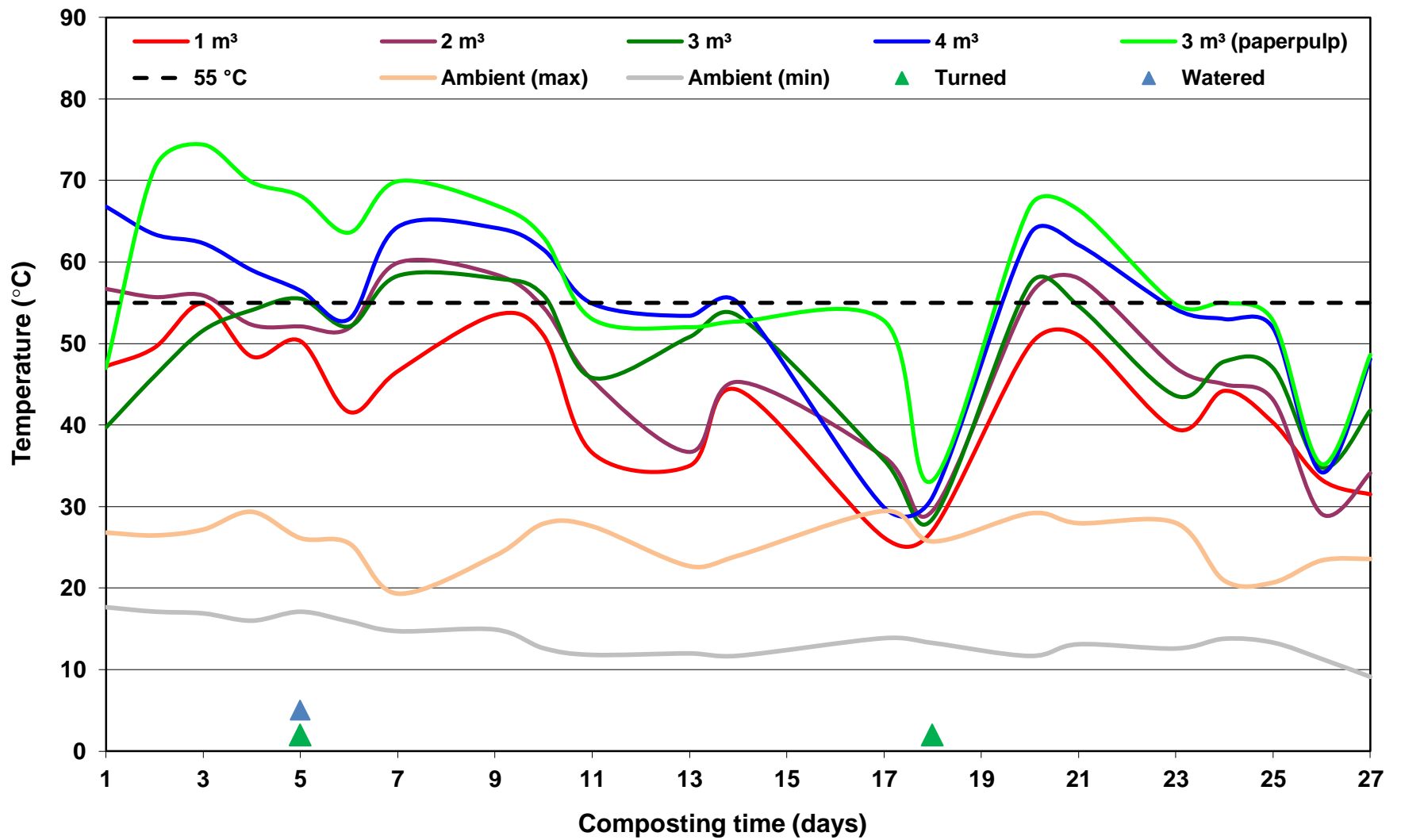
Figure 3.2 Core temperatures of piles of different sizes measured over 27 days of composting during the preliminary trial
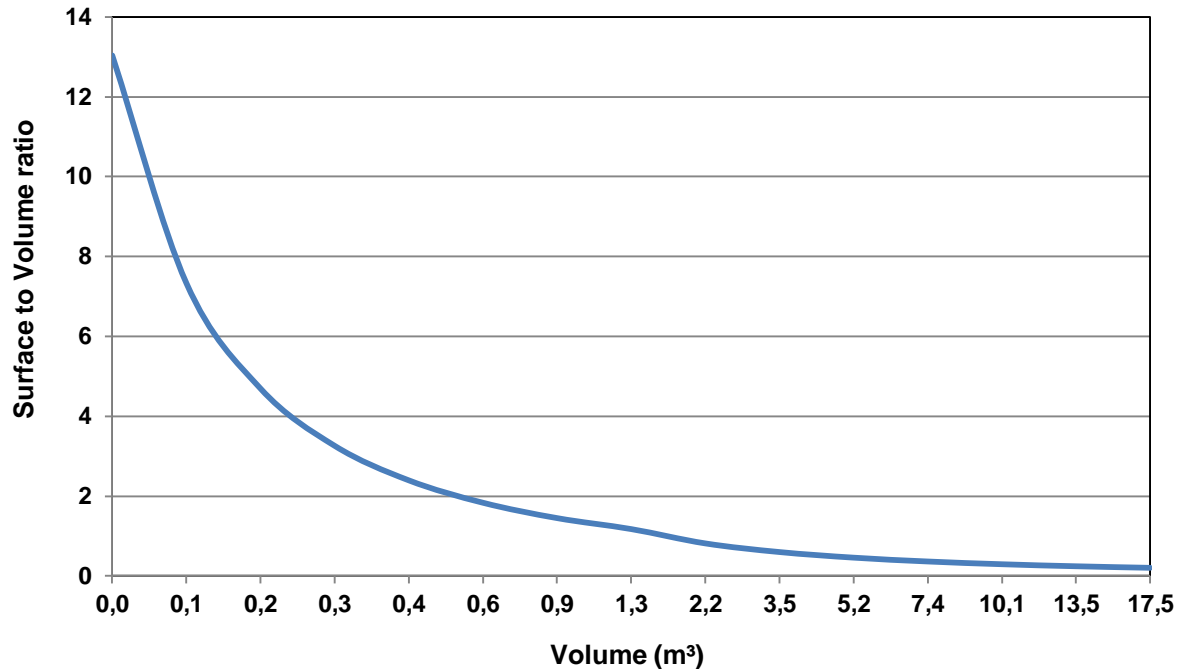
Figure 3.3 The exponential relationship between the surface-to-volume ratio and the volume of a compost pile (calculated for a cone shape) indicates that a high surface-to-volume ratio, which can logically be expected to have a negative effect on pile temperature, occurs in low pile volumes but rapidly declines with increasing pile volume and can be expected to be low enough for pile volumes of around 4 m^3 and larger, not to impact negatively on pile temperature

Another explanation could be the exothermic dissolution/neutralisation reaction involving $CaCO_3$. For example if HCO_3^- is assumed to be the dominant species and considering the net reaction:

$$CaCO_{3(s)} + H^+_{(aq)} \rightleftharpoons Ca^{2+}_{(aq)} + HCO3^-_{(aq)}$$
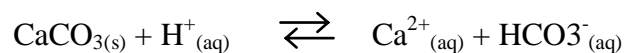
From the standard enthalpy of formation for the species (Stumm and Morgan, 1996), the enthalpy of the reaction is calculated as:

The sum of the enthalpy of the products:

$[Ca^{2+}_{(aq)} + HCO3^-_{(aq)}] = [(-542.8) + (-692.0)]$ $\qquad = \qquad$ -1234.8 kJ.mol^{-1}

47

Less the sum of the enthalpy of the reagents:

$[CaCO_{3(s)} + H^+_{(aq)}] = [(-1207.4) + (0)]$ = (-1207.4) kJ.mol^{-1}

Equals: Enthalpy of the reaction (exothermic) -27.4 kJ.mol^{-1}

From Chapter 1, the heat of combustion for sewage sludge can be expected to be about 23.2 kJ.g^{-1} dry volatile solids (Vesilind, 1979) and although exothermic, the energy release from $CaCO_3$ (27.4 kJ.mol^{-1} or 0.27 kJ.g^{-1}) is two orders of magnitude less per gram. The mass of $CaCO_3$ at an estimated 4 to 5% (from an indicative 45% in the paper pulp and a 10% dilution in the mixture) is about a quarter of the expected dry volatile solids "from sludge" content of the organic (sawdust-sludge) component of the pile of roughly 20% (80 to 90% dry volatile solids of which roughly 20% is from sludge – if a quick mass balance of solids and volatile solids contents is performed). Reaction kinetics would be expected to play an important role in the heating effect but the observed higher temperature more likely resulted from the paper pulp being a more readily degradable source of C and available Ca, the higher available water content and/or to a more favourable pH for microbial activity.

Valuable observations made during the preliminary phase trial that contributed to the design of the main composting trial were:

- The sludge-sawdust mixture resulted in a feedstock with adequate porosity and water holding capacity (from sense observations).
- Dewatered sludge tends to clump into small aggregates when mixed with the much drier sawdust and the distribution of aggregates throughout the pile was good.
- Absorbed water contained in dewatered sludge is at a lower energy state than free water added to a pile - resulting in a slower water release rate and slower drying.
- Bigger piles that have lower surface-to-mass-ratios reach higher temperatures and are more buffered against cooling due to low ambient temperatures and wind. This relationship can be expected to diminish rapidly and become insignificant in piles larger than 4 m^3.

- The higher temperature observed in the pile to which paper pulp was added, most likely resulted from the paper pulp being a more readily degradable source of C and available Ca, the higher available water content and/or to a more favourable pH for microbial activity. The paper pulp could also have altered the thermal capacity of the feedstock/pile.

- The highest temperature in a particular pile was measured at ⅓ of the pile height above the ground.

- The temperature measurements stabilised sufficiently after leaving the temperature probe in the pile to reach equilibrium for 5 minutes (refer to Figures 3.6 and 3.7 below).

3.2.2 Main composting trial

In the main composting trial, three types of WWS (Figures 3.4 and 3.5) were composted with Eucalyptus sawdust as bulking agent and a small amount of highly reactive dolomitic lime as amendment to compensate for the slightly acidic sawdust (pH 5). These were:

- Dewatered, activated sludge from an industrial (petrochemical) water treatment plant (ACT-IND);

- Dewatered, activated sludge from a municipal water treatment plant (ACT-MUN); and

- Evaporation dried, anaerobically digested sludge from a municipal water treatment plant (DIG-MUN).

Mixture formulation was primarily based on the gravimetric water content of raw materials while also considering their carbon and nitrogen content, specifically the C:N ratio (see Appendix A for calculation details). Initial calculations for mixture formulation were based on assumed or expected water content for the different WWS (as obtained from discussion with the respective water treatment plant managers) and carbon and nitrogen contents measured in samples previously taken from the same sources. These samples have been stored and could therefore not be utilised to determine the water content of the WWS.

Figure 3.4 Centrifuge dewatered activated sludge from an industrial water treatment plant at SASOL Secunda (ACT-IND, left) and belt pressed dewatered activated sludge from a municipal water treatment plant at ERWAT Olifantsfontein (ACT-MUN, right)

Figure 3.5 Evaporation dried anaerobically digested sludge (left) and sludge collection on site at ERWAT Vlakplaats municipal water treatment plant showing anaerobic digesters, activation dams, and drying pans in the background (DIG-MUN, right)

Some variation (temporal) was expected between the characteristics of these samples and the actual materials used, but given the fact that composting is reported to proceed effectively over a range of chemical and physical conditions, this was considered adequate for calculation of mixing ratios. The expected and measured characteristics of the samples as well as the calculated mixing ratios and feedstock characteristics are presented in Table 3.1.

Table 3.1 Raw material characteristics, target water content and calculated feedstock requirements (amounts) and characteristics (C:N)

Raw material characteristics					Calculated feedstock requirements		Target feedstock water content and resultant C:N	
Material	Water[1] %	C %	N %	C:N ratio	Wet mass (kg)	Water (kg)	Water %	C:N ratio
ACT-IND (P1)	91	39.0	7.91	4.93	681	0	**63.0**	26.0
DIG-MUN (P2)	40	11.6	1.93	5.99	875	1059	**63.0**	18.0
ACT-MUN (P3)	82	29.9	5.33	5.61	1003	0	**63.0**	18.2
ACT-MUN (P4)	82	29.9	5.33	5.61	1300	0	**66.0**	15.8
SAWDUST	30	40.9	0.61	67.0	**580**	-	-	-

1: Water content of sludge based on average reported by respective WCW

The wet bulk density of the sawdust was measured by determining the mass of 25 litre samples and the average of 145 kg.m^3 meant that the mass of a 4 m^3 pile would be 580 kg. The initial target water content for the WWS-sawdust mixtures was 63% (from literature reviewed) and a fourth treatment consisting of WWS$_{ACT-MUN}$ was included to test the effect of a 30% higher sludge load.

Fresh samples of WWS were taken from the respective water treatment plants shortly before the actual batches of materials to be used in the trial were collected, and their water content was determined. These were incorporated into the calculations while keeping the previously calculated mixture masses the same (Table 3.2). It was decided to only adjust the required amount of water because the feedstock water content and C:N ratios were within acceptable limits.

Table 3.2 Material characteristics (with adjusted water content), target feedstock amounts and calculated water requirements and feedstock characteristics

Raw material characteristics					Target feedstock amounts and calculated water requirements		Calculated feedstock characteristics	
Material	Water %	C %	N %	C:N ratio	Wet mass (kg)	Water (kg)	Water %	C:N ratio
ACT-IND (P1)	87.1	39.0	7.91	4.93	**681**	76	63.0	21.3
DIG-MUN (P2)	50.7	11.6	1.93	5.99	**875**	810	63.0	20.1
ACT-MUN (P3)	85.1	29.9	5.33	5.61	**1000**	0	64.8	20.3
ACT-MUN (P4)	85.1	29.9	5.33	5.61	**1300**	0	68.1	17.6
SAWDUST	29.8	40.9	0.61	67.0	**580**	-	-	-

Volumetric measurements were employed to determine the raw material amounts. This was done as accurately as possible by determining the wet bulk density of the raw materials through determining the mass of fixed volume samples and then calculating the respective volumes of materials required (Table 3.3).

The density of dewatered WWS was assumed to be 1000 kg.m^3 (from discussions with WWS laboratory officials). Volumetric measurements of the raw materials were achieved by filling a cylindrical container to a specific calculated height. Sawdust piles of 4 m^3 were measured off on a roofed concrete floor. The calculated amounts (volume) of sludge

52

for each treatment were measured off and added to the respective sawdust piles. Feedstock was thoroughly mixed by hand and tap water was added during mixing where required.

Table 3.3 Raw material physical parameters

Material	Calculated wet mass required (kg)[1]	Wet bulk density (kg.m^{-3})	Volume used (m^3)
ACT-IND (P1)	680	1000	0.68
DIG-MUN (P2)	880	786	1.12
ACT-MUN (P3)	1000	1000	1.00
ACT-MUN (P4)	1300	1000	1.30
SAWDUST	580	145	4.00

1: Rounded off to the nearest ten

P1 was visibly drier than the other treatments after receiving its 76 litres of water and a total of 180 litres of water was added to this pile. The addition of initially required water to P2 (810 litres) had to be done on three separate occasions to prevent leaching and hand mixing had to be performed 2 to 3 times for all piles to effectively reduce the size of sludge clumps and achieve relatively homogenous mixtures. Tap water was added with a hose pipe equipped with a water flow meter.

Composting and monitoring of piles started around the middle of June 2006 and due to the low ambient temperatures and cold wind, piles were covered with plastic sheeting from day 17 to prevent excessive cooling from wind. The covering of compost piles with plastic sheeting could reasonably be expected to affect the natural ventilation and gas exchange of compost piles with the environment but was found to be absolutely necessary to allow for reasonable control of the environmental conditions of compost piles.

Piles were manually turned, sampled and watered at different time intervals during composting. The amount of water added to compost piles throughout the composting process was judged from the look of individual piles when turned. Pile temperature was measured and recorded with a one meter long thermocouple probe (K-type) with four thermocouples at 25 cm intervals and digital thermometer/data logger (YC-741D) with RS-232 interface with windows software (Figure 3.6).
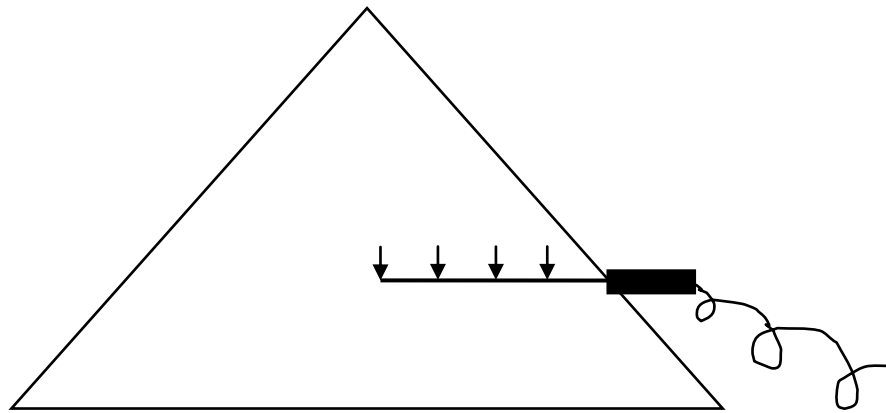
Figure 3.6 Diagram depicting the approximate placement of the temperature probe (1 meter in length) in compost piles and the position of the thermocouples in the probe (arrows) during temperature measurement

Through trial and error it was determined that the maximum pile temperature (assumed as the core temperature) is measured at a height of about ⅓ of the pile height and in almost all cases recorded by the thermocouple located at the point of the probe i.e. one meter into the pile. An equilibration time of five minutes was found to be more than adequate for reading stabilisation before the measurement was taken. This protocol was applied throughout the study. Ambient temperature data was obtained from the automatic weather station located at the University of Pretoria Hatfield Experimental Farm.

Water content of raw materials and compost piles was measured gravimetrically after oven drying of samples (3 replicates) at 100°C for 24 hours (70°C for sawdust). Bulk density of raw materials and compost mixtures was measured through weighing of a

54

fixed volume or was assumed to be 1000 kg.m^3 (from discussions with WWS laboratory officials). The volume of compost piles was approximated from the volume of a cone shape and calculated from measurements of pile height and radius after piles were turned and reconstructed (Figure 3.7).

Figure 3.7 The 4 m^3 piles after initial mixing (left) and recording of pile temperature (right)

Laboratory analyses of raw material, feedstock and compost samples (3 replicates) consisted of the following:

- Total C and N content were determined with a Carlo Erba NA 1500 C/N/S Analyzer (see Appendix A for detail).
- Potentiometric pH was measured in a 1:10 (mass base) water extract after equilibrating for 24 hours (stirred initially when mixed and before pH was measured) using a pH electrode and pH meter calibrated against buffers at pH 4 and pH 7 (Thompson *et al*., 2002).
- Electrical conductivity (EC) was measured with a suitable conductivity electrode and meter in the same water extract as pH (Thompson *et al*., 2002).
- Ash/volatile solids content was measured gravimetrically after combustion of oven dry samples at 550 °C for 4 hours in a muffle furnace (Thompson *et al*., 2002).

Statistical analysis of data was performed with SAS 9.1 and Microsoft EXCEL.

55

3.3 Results and discussion

The results are discussed in terms of the measured characteristics of actual batches of raw materials used, the measured characteristics of feedstock at the start of composting and the evolution of certain measured parameters during the composting process.

3.3.1 Raw materials

The measured characteristics of the raw materials (bathes actually used in the main trial) appear in Table 3.4. The water content of dewatered sludge (ACT-IND, ACT-MUN) was relatively high and probably represents the lower limit typically achieved with the addition of an electrolyte (flocculation agent) followed by mechanical dewatering (centrifuge or belt press). From a composting perspective the high water content necessitates the addition of a substantial amount of suitable bulking agent while reducing the need for additional input of water. Evaporative drying of sludge takes time, space and frequent turning but much lower water content can be achieved (DIG-MUN).

Table 3.4 Measured raw material characteristics (± standard deviation – three replicates)

Material	Water (%) wet mass	C (%) dry mass	N (%) dry mass	C:N Ratio	pH[1]	EC[1] (mS.m^{-1})	Ash[2] (%) dry mass
ACT-IND (P1)	88.9 ±0.03	53.1 ±0.18	10.0 ±0.03	5.30 ±0.03	6.20 ±0.02	356 ±28.6	8.90 ±0.16
DIG-MUN (P2)	52.3 ±0.06	19.2 ±0.43	2.49 ±0.03	7.73 ±0.23	4.36 ±0.03	474 ±11.7	55.6 ±0.14
ACT-MUN (P3)	87.2 ±0.07	37.7 ±1.15	7.03 ±0.20	5.36 ±0.03	6.01 ±0.03	498 ±24.6	27.3 ±0.16
ACT-MUN (P4)	86.4 ±0.21	39.1 ±0.18	7.33 ±0.07	5.33 ±0.04	6.35 ±0.09	526 ±28.3	27.3 ±0.03
SAWDUST	30.9 ±4.91	47.2 ±0.17	0.21 ±0.02	221 ±16.2	5.05 ±0.04	23.0 ±3.46	2.62 ±0.41

1: air dry, milled samples – water content taken into account for 1:10 (mass base) water extract; 2: oven dry, milled samples

The anaerobically digested sludge (DIG-MUN) had a slightly higher C:N ratio than the activated sludge but had substantially lower contents of C and N. These elements are lost as CH_4 and NH_3 gas during digestion, leaving the remaining sludge depleted in organic matter, as reflected by its high ash content (or its reciprocal low volatile solids content).

Soil contamination from handling on a bare soil surface and contamination from windblown dust in drying basins at the WCW can be expected to be partly responsible for the high ash content of this sludge. Published data on anaerobically digested sludge indicate pH values near neutral (Haug, 1993) and leads one to attribute its low pH of 4.36 to chemical reactions that occurred during evaporative drying and simultaneous further stabilisation, such as the oxidation of reduced compounds including organic C and N and especially, nitrification of ammonium.

These deductions are supported by a parallel study on nitrogen mineralisation in sludge amended soils at the Department of Plant Production and Soil Science, University of Pretoria, that characterised the same three types of WWS as used here (Nobela, 2011). Apart from also reporting substantially lower total N content for DIG-MUN sludge (1.93%) compared to ACT-IND (7.91%) and ACT-MUN sludge (5.33%), they also reported the distribution of N forms in the sludge (Table 3.5).

Table 3.5 The percent distribution of N-forms in the sludge – reproduced (Nobela, 2011)

Source of sludge	NH_4^+ (% of total N)	NO_3^- (% of total N)	Organic N (% of total N)
SASOL (ACT-IND)	4.62	0	93.9
VLAKPLAAS (DIG-MUN)	26.6	28.0	53.4
OLIFANTSFONTEIN (ACT-MUN)	1.75	0	97.6

The data show that a substantial amount of N mineralisation occurred in the DIG-MUN sludge and that it only contained 53.4% organic N compared to 93.9% and 97.6% in the

57

ACT-IND and ACT-MUN sludge respectively. Furthermore, 28.0% of the N in the DIG-MUN sludge was already in the nitrate form, meaning that roughly 50% of the mineralised N has undergone nitrification while no nitrification occurred in the ACT-IND and ACT-MUN sludge.

EC is a qualitative measure of dissolved salts (e.g. NH_4^+, NO_3^-, SO_4^{2-} etc.) that can be the result of organic matter mineralization or salts occurring in or added to the organic material. High EC values could be seen to represent advanced organic matter mineralisation and by deduction, lower organic matter content but from the data presented in Table 3.4 and Table 3.5 it is clear that this is not necessarily a correct assumption and that total C or ash content (or its reciprocal volatile solids content) would be more suitable as quantitative indicators of organic matter content although it should be taken into account that ash content could be largely inflated where a risk of soil contamination exist (e.g. DIG-MUN).

The sawdust was fairly dry, contained mostly organic carbon and was somewhat acidic with a pH of 5.05 measured in air dried, milled samples (Table 3.4) and pH of 4.45 measured in oven dried un-milled samples. It was decided to add dolomitic lime to the composting mixtures in order to increase the pH.

To determine the amount of lime needed, a pH buffer experiment was performed by adding incremental amounts of a saturated (0.02 M) $Ca(OH)_2$ solution to a 20 g sawdust : 200 ml distilled water suspension (oven dried un-milled sawdust samples measuring pH 4.45, Figure 3.8).

The pH of the suspension increased almost linearly with increasing amounts of $Ca(OH)_2$ solution up to around pH 5.5 where after pH increase was slightly buffered. The distilled water (control) measured a pH of 6.1 and it was decided to neutralisation to this pH (0.5 mmol $Ca(OH)_2$ added with 25 ml of 0.02 M $Ca(OH)_2$). This meant that 2.5×10^{-2} mol $Ca(OH)_2$ was required per kg sawdust.

58
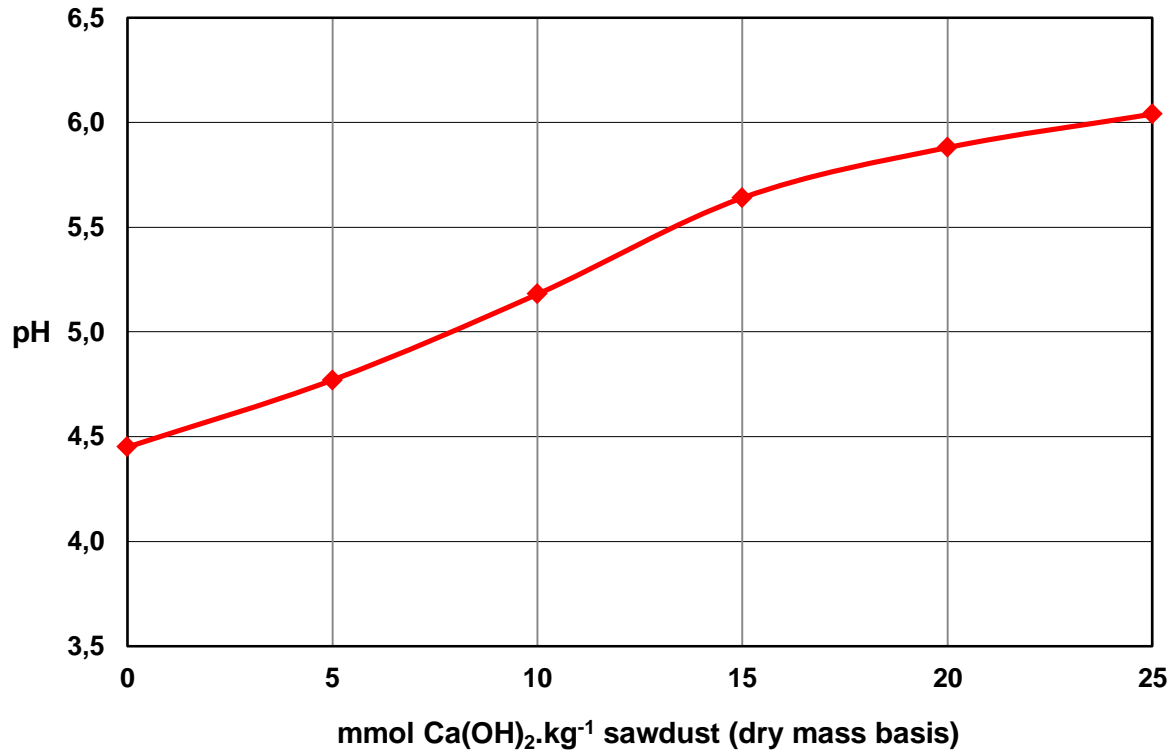
Figure 3.8 The pH response-curve to determine the amount of lime needed to neutralise sawdust in compost piles

The composition of the lime was not known and it was assumed to be 100% pure and to contain 50% calcite ($CaCO_3$) and 50% dolomite ($Ca_{(0.5)}Mg_{(0.5)}CO_3$). From the molar masses of $CaCO_3$ (100g.mol^{-1}) and $Ca_{(0.5)}Mg_{(0.5)}CO_3$ (92g.mol^{-1}) the molar mass for the lime used was calculated to be 96g.mol^{-1}. This equated to 2.40 g lime per kg sawdust or 1.39 kg lime per 4 m^3 (580 kg) sawdust. The lime was extremely fine and assumed to be 100% reactive. No steps were taken to adjust the pH further in the treatment receiving the relatively acidic (pH 4.36) DIG-MUN sludge.

A study by Wong and Fang, 2000 performed in a bench reactor, showed that the co-composting of WWS and lime effectively reduced the availability of heavy metals in WWS compost. It was found that lime raised the pH of a sludge/sawdust feedstock effectively during the initial period and the effect became less obvious with an increase in composting time. Adverse effects of lime amendment on all biological parameters tested (microbial respiration, microbial populations, and activities of related enzymes) increased

59

with an increase in lime application rates, but these effects were generally restricted to the early stage of the thermophilic phase. Addition of 0.63% (dry mass basis) lime prior to sludge composting slightly improved the microbial activity as indicated by the higher temperature and CO_2 evolution and did not exert a significant inhibition on bacterial population, and the activities of related enzymes after 100 days of composting.

The high alkalinity of lime was the major reason for the reduction of microbial and enzyme activities following lime amendment and the addition of lime > 0.63% (dry mass basis) would affect the decomposition of sewage sludge and extend the time required for maturation. This was supported by a longer thermophilic phase for the treatments with 1.0 and 1.63% (dry mass basis) lime amendment. The microbial and enzyme activities were also slightly lower in the mature compost with 1.0 and 1.63% lime amendment (Wong and Fang, 2000).

3.3.2 Feedstock characteristics

The WWS to sawdust (bulking agent) ratios, as well as the amounts of free water added to each of the four treatments, are presented in Table 3.6.

Table 3.6 WWS to bulking agent ratios of treatments (initial compost piles)

Compost Pile	WWS : sawdust (wet mass)	WWS : sawdust (volume)	Water added (litres)
P1$_{ACT-IND}$	1.2 : 1	1 : 5.9	180
P2$_{DIG-MUN}$	1.5 : 1	1 : 3.6	810
P3$_{ACT-MUN}$	1.7 : 1	1 : 4.0	0
P4$_{ACT-MUN}$	2.2 : 1	1 : 3.1	0

Once the actual batches of raw materials to be used in the main trial had been analysed, the expected feedstock characteristics when combining the pre-determined feedstock amounts, could be re-calculated and these are presented in Table 3.7.

Table 3.7 Measured raw material characteristics, actual feedstock amounts used and calculated (expected) feedstock characteristics

Measured raw material characteristics					Actual feedstock amounts		Calculated feedstock characteristics	
Material	Water %	C %	N %	C:N ratio	Wet mass (kg)	Water (kg)	Water %	C:N ratio
ACT-IND (P1)	88.9	53.1	10.0	5.30	680	180	66.9	27.3
DIG-MUN (P2)	52.3	19.2	2.49	7.73	880	810	63.9	23.9
ACT-MUN (P3)	87.2	37.7	7.03	5.36	1000	0	66.5	24.1
ACT-MUN (P4)	86.4	39.1	7.33	5.33	1300	0	69.3	18.7
SAWDUST	30.9	47.2	0.21	221	580	-	-	-

As previously mentioned, the addition of initially required water to P2 (810 litres) had to be done on three separate occasions to prevent leaching and hand mixing had to be performed 2 to 3 times for all compost piles to effectively reduce the size of sludge clumps and achieve relatively homogenous mixtures. Therefore, the initial mixture characteristics of the four treatments shown in Table 3.8 represent data from samples taken on day 9 after materials were first combined and formed into piles.

After the addition of 180 litres of free water to P1 the mixture still looked a bit dry and this was evidenced by a measured gravimetric water content of 60.0% (expected to be 66.9%). Considering this and its measured C:N ratio of 29.7 (expected to be 27.4) this treatment could have received more ACT-IND sludge.

The pH of P1 (6.83) was higher than that of the ACT-IND sludge (6.20) and the saw dust (5.05) in the feedstock and could be evidence to the effect of the added lime. It's low EC (56.0 mS.m^{-1}) and ash content (10.2%) supported the low mineralisation (high organic matter) status of the ACT-IND sludge and treatment P1.

61

Table 3.8 Measured feedstock characteristics (± standard deviation – three replicates)

Compost Pile	Water (%) wet mass	C (%)	N (%)	C:N Ratio	pH[1]	EC[1] (mS.m⁻¹)	Ash[2] (%) dry mass
		Dry mass					
P1$_{ACT-IND}$	60.0 ±4.19	42.0 ±1.15	1.43 ±0.14	29.7 ±3.15	6.83 ±0.18	56.0 ±16.7	10.2 ±0.24
P2$_{DIG-MUN}$	54.7 ±0.49	24.0 ±0.27	1.24 ±0.02	19.3 ±0.48	4.88 ±0.01	287 ±56.2	26.3 ±0.20
P3$_{ACT-MUN}$	61.9 ±1.40	44.5 ±0.35	1.44 ±0.08	30.9 ±1.74	7.59 ±0.11	253 ±22.9	12.7 ±0.16
P4$_{ACT-MUN}$	67.4 ±2.10	38.2 ± 0.48	1.89 ±0.17	20.3 ±1.91	7.41 ±0.04	211 ±52.2	12.4 ±0.34

1: as sampled – water content taken into account for 1:10 (mass base) water extract

2: oven dry, milled samples

After receiving 810 litres of free water, P2 leached slightly and the measured water content of 54.7% (expected to be 63.9%) is representative of the water holding capacity of this mixture. The measured C:N ratio of 19.3 was lower than the expected C:N ratio of 23.9 and the pH of 4.88 was higher than the 4.36 of the DIG-MUN sludge but still lower than the pH of 5.05 of the sawdust bulking agent and seems to attest to a rather limited effect of the pH correcting addition of lime.

The low C content, low pH, and high ash content of the DIG-MUN sludge is reflected in these characteristics measured in P2 and together with a relatively high EC (287 mS.m⁻¹) is reliable evidence of the low organic matter (or high mineralisation) status of the DIG-MUN sludge and therefore of treatment P2.

No initial free water was added to P3 and P4 and a comparison of these two treatments clearly illustrate how a higher dewatered sludge to sawdust ratio will be limited by a too high feedstock water content and/or a too low feedstock C:N ratio when these parameters reach the limits for reasonable composting conditions (Table 1.1, p.8). Similar to treatment P1, a trend of lower than expected water content for P3 of 61.9% (expected to

62

be 66.5%) and P4 of 67.4% (expected to be 69.3%) and higher than expected C:N ratios of 30.9 for P3 (expected to be 24.1) and 20.3 for P4 (expected to be 18.7) was observed.

The pH values of 7.59 (P3) and 7.41 (P4) were higher than the 6.01 and 6.35 measured in the respective batches of ACT-MUN sludge and seems to be the effect of the pH correcting addition of lime. The EC of P3 (253 mS.m^{-1}) and P4 (211 mS.m^{-1}) was substantially higher than that of P1 (56.0 mS.m^{-1}) but not as high as that of P2 (287 mS.m^{-1}) and the ash content of P3 (12.7%) and P4 (12.4%) was substantially lower than that of P2 (26.3%) but higher than that of P1 (10.2%). This indicates that although P3 and P4 contained a fair amount of dissolved salts, they were roughly equally rich in organic matter as P1 (further supported by comparable C and N contents).

3.3.3 Composting process

The core temperature of compost piles (Figure 3.9) showed large and frequent variations during the first 17 days due to turning, addition of water (P1, P3) and winter temperatures. Some very high initial temperatures were recorded for P3 (70°C) and P4 (73°C) during this period but it was evident that the piles were not well buffered against cooling due to low ambient temperatures and especially, cold wind. Piles were covered with plastic sheeting on day 17 and left undisturbed for about a month (Figure 3.10).

Pile temperature stabilised at sustained high levels with smaller fluctuations and a general pattern of P1 > P4 > P3 > P2. Temperature started dropping off towards the end of this period and piles were turned (no water added) and reconstructed on day 45. Recovery after turning was very good and piles returned and even exceeded their former high temperature levels.

A very cold night with strong wind on day 53 saw the plastic sheeting blown of P1, P2 and P3 while P4 was still covered. Temperature of the exposed piles plummeted while P4 only dropped 6°C to 47°C and soon recovered to above 50°C, illustrating the effect of cold wind and the necessity to cover the piles. Again temperature recovery was very good and turning and water addition to all piles on day 61 seemed to enhance this process.

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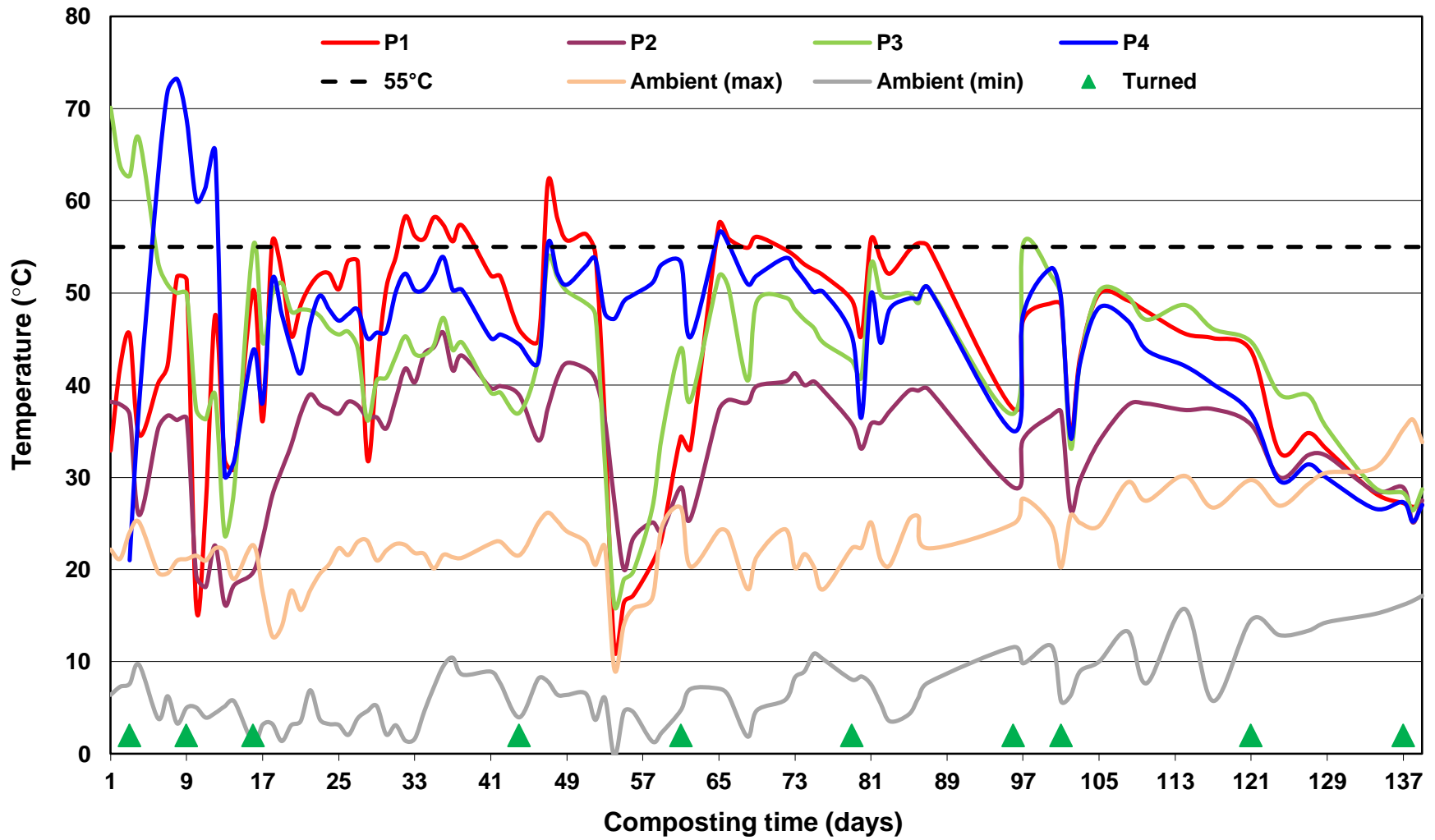
Figure 3.9 Core temperature of compost piles, minimum and maximum air temperature and turning incidence over 139 days of composting

Figure 3.10 Compost piles were covered with plastic sheeting to withstand the cooling effect of winter temperatures and cold wind (picture taken towards the end of the process - the differences in final pile sizes are clearly noticeable with P1 on the far left and P4 on the far right)

This time the period of sustained high temperature lasted roughly half the length of time as previously and was on a steady downward trend when piles were mixed and water added on day 79. Pile temperature recovered well but an unfortunate loss of data from day 88 to 95 makes the period of sustained high temperature uncertain.

Piles were mixed and watered on day 96 and again mixed for sampling on day 101 when, based on the temperature trend at the time, it was clear that the active (thermophilic) phase of composting was coming to an end (for P1, P3 and P4 with P2 never really reaching sustained thermophilic temperatures). Thermophilic temperature is generally considered to be above 45°C (Brady and Weil, 1999) with maximal thermophilic activity occurring between 52°C and 63°C (Atlas and Bartha, 1998).

After day 101 pile temperature did exceed 45°C (P1, P3 and P4) but did not recover to above 50°C after turning and the day 101 samples were deemed representative of the end of the thermophilic phase. Over the next 38 days of curing, pile temperature gradually declined in spite of turning and water addition on days 121 and 137. The composting

65

process was deemed over when pile temperature did not recover to above day time ambient temperature (Figure 3.11).

Figure 3.11 Compost piles at the end of the curing phase (final product)

Certain measured process parameters are presented in Table 3.9. The highest core temperature for P3 and P4 was recorded in the initial stages of composting, indicating a high initial energy value that can be ascribed to easily degradable, volatile organics (e.g. volatile fatty acids) in the ACT-MUN sludge (from discussions with WWS laboratory manager).

P1 showed a somewhat different trend in that its temperature increased more conservatively yet steadily in the initial stages and that its highest core temperature was only recorded on day 47. Considering the minimum amount of time (only days actually recorded and therefore not interpolated) that pile core temperature exceeded 50°C and 55°C (Table 3.9), P1 showed the most sustained heat production, followed by P4 and then P3.

66

Table 3.9 Process parameters

Compost Pile	Maximum core Temperature	Days > 50° C	Days > 55° C	Water content range (%)	Volume reduction (%)
P1$_{ACT-IND}$	62	43	21	54-71	53
P2$_{DIG-MUN}$	46	0	0	50-59	17
P3$_{ACT-MUN}$	70	27	5	57-67	49
P4$_{ACT-MUN}$	73	38	10	58-67	49

This can be ascribed to more energy released from its higher VS content and/or the more polymerised and aromatic nature of organic compounds in the ACT-IND sludge (from discussions with SASOL environmental scientist). The maximum temperature recorded for P2 was 46°C confirming that paddy-dried, digested sludge (DIG-MUN) is relatively stable and has a lower energy value compared to activated sludge.

Required water addition to piles during composting was judged from the look and feel of mixtures when piles were turned. The first measurement for pile water content was made on day nine after the addition of the initial required feedstock mixture water to P1 and P2 (Figure 3.12 and 3.13). P1 appeared dry and on day 16 water was added to P1 (130 litres) and all piles were turned. Piles were left undisturbed until day 45 when they were turned, sampled and water content determined. Over this time the water content of piles declined to between 50 and 58% (wet mass). On day 61, piles were turned and water was added to all piles before the water content was measured. On day 79, the water content was measured before water was added to P1, P3 and P4. At this stage the material of all piles looked sufficiently wet and only small amounts or no water was added. Water was added in increasing amounts to all piles on days 95, 121 and 137 and water content was measured after water addition and turning. This trend can be attributable to the increase in atmospheric demand with ambient temperatures steadily increasing over this period to its maximum for the entire composting period of 36 ºC (day time) and 16 ºC (night time) on day 137 (Figure 3.9).

67
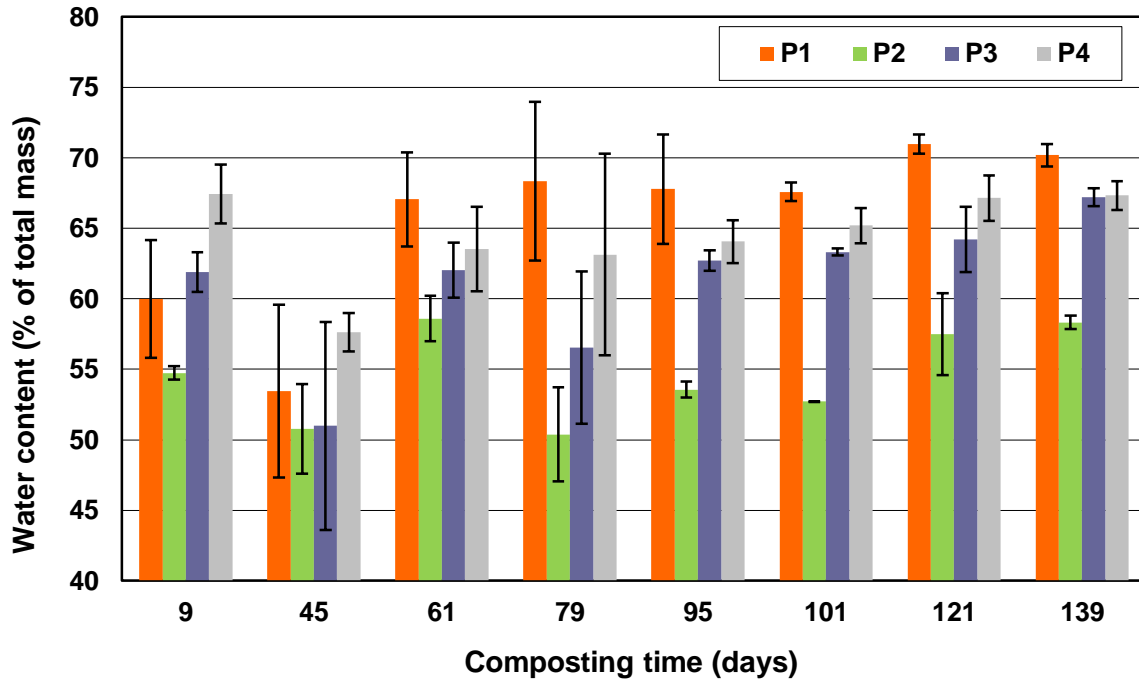
Figure 3.12 Gravimetric water content of compost piles at times of measurement (error bars represent standard deviations – three replicates)
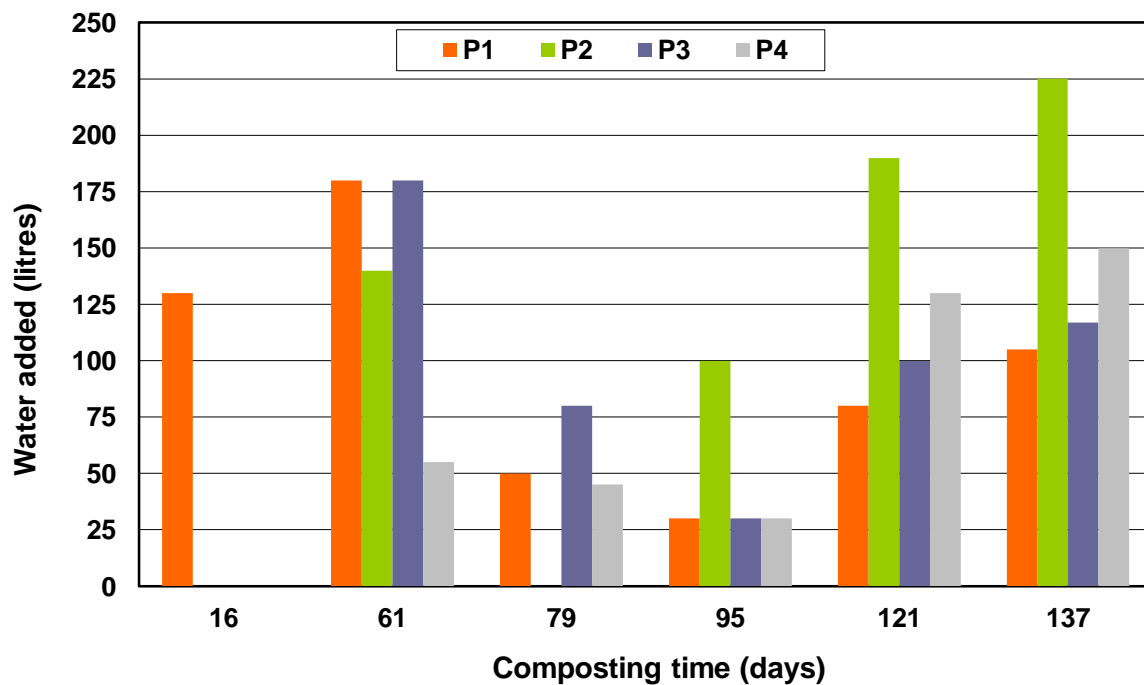
Figure 3.13 Water added to piles during composting (excluding the initial feedstock mixture's water requirement)

68

The recorded water content of piles was between 50 and 71% (wet mass) over the entire composting period and the total amount of water added (excluding the initial feedstock mixtures water requirement) over 139 days of composting was 575 litres (P1), 655 litres (P2), 507 litres (P3) and 410 litres (P4) respectively. The high water content of P1 during the curing phase could have contributed to its temperature decline due to the large heat capacity of water.

The covering of compost piles with plastic sheeting would have affected the evaporation of water from the piles thus decreasing water losses to the atmosphere and lowering the amount of water to be added. Also, through condensation, the water distribution in compost piles was affected and resulted in water accumulation in the upper part and interestingly, the south-facing surface, of piles (Figure 3.14).

Figure 3.14 The effect of covering compost piles with plastic sheeting on the water distribution in P1$_{\text{ACT-IND}}$

Measurements of pile volume were made after piles were turned and reconstructed and appear in Figure 3.15.
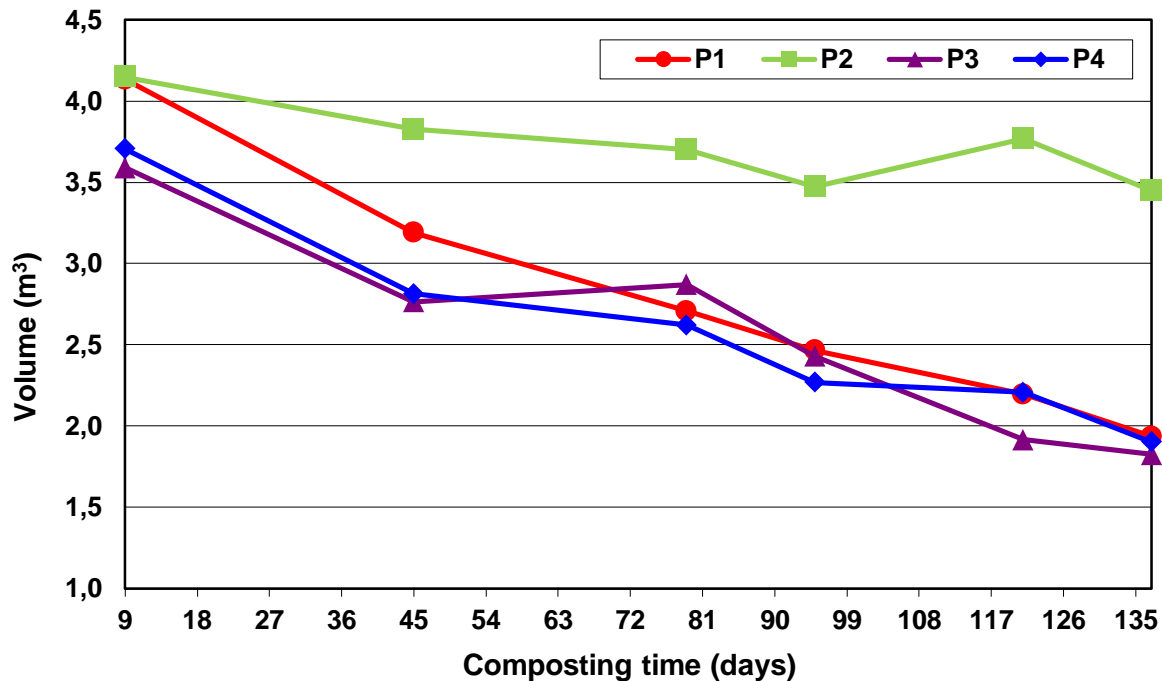
Figure 3.15 Change in pile volume over 135 (out of 139) days of composting

The final reduction in pile volume was similar for P1 (53.2%), P3 (49.1%) and P4 (48.7%) while a far smaller reduction occurred in P2 (16.8%). Initial pile volumes were close to the 4 m^3 starting volume of sawdust. The lower initial volumes of P3 and P4 seems to be representative of compaction due to the higher wet mass of sludge and resultant higher initial water content of the piles. This was also clearly observed from their size (especially height, almost slumping) before turning and reconstruction i.e. after standing undisturbed for some time. This would imply a reduced air filled porosity (free air space) and possible lower ventilation rate in these piles which could explain why their core temperature, after initial highs during the first two weeks, was consistently lower than that of P1 up to day 95 (Figure 3.9). Although volume measurements were rather crude, changes in pile volume were consistent with and supported temperature observations and interpretations.

Wet bulk density measurements are dependent on pile water content at the time of measurement and were adjusted for water content to obtain dry bulk density (Figure 3.16). Initial feedstock dry bulk densities for P1 (110 kg.m^{-3}), P3 (114 kg.m^{-3}) and P4

70

(113 kg.m^{-3}) were similar and again showed an equally similar pattern of decline over the active phase of composting and increasing over the curing phase with final dry bulk densities for P1 (114 kg.m^{-3}, 3.36% increase), P3 (121 kg.m^{-3}, 6.36% increase) and P4 (133 kg.m^{-3}, 18.1% increase) being higher than that of the initial feedstock and the % increase reflecting the increasing sludge content of these piles.

P2 had an initial feedstock dry bulk density (224 kg.m^{-3}) that was roughly double that of the other piles (more evidence of possible soil contamination) and which also declined over the active phase and increased over the curing phase, but its final dry bulk density (173 kg.m^{-3}, 22.9% decrease) was lower than that of its initial feedstock.
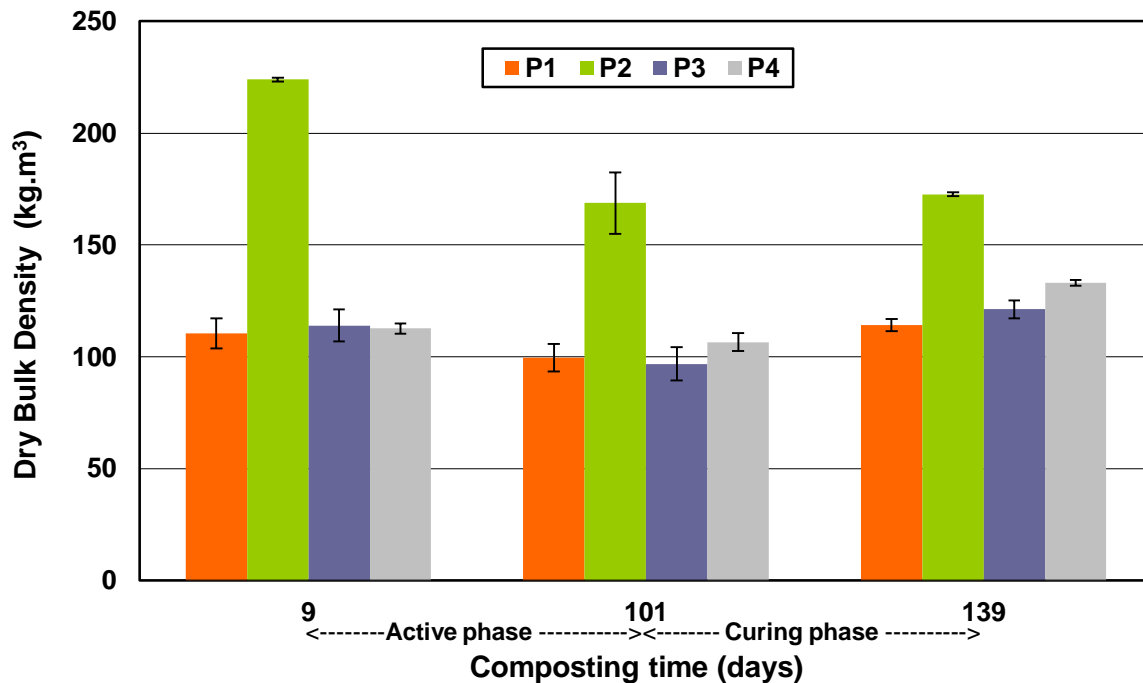
Figure 3.16 Change in dry bulk density of compost piles over 139 days of composting and specifically after the active and curing phases (error bars represent standard deviations - three replicates)

Lastly, the manual mixing of dewatered WWS with the sawdust resulted in the formation of "sludge aggregates" (Figure 3.17) which diminished in size and amount with manual turning but, nevertheless, persisted till the end of the composting period.

71

Figure 3.17 Example of "sludge aggregates" that formed when dewatered WWS and sawdust were mixed for composting feedstock – also showing the white hyphae of decomposing fungi (right)

These "aggregates" did allow for some interesting qualitative observations when broken observing the inside after 139 days of composting (Figure 3.18). Those from P1 were black in colour and almost rubbery in consistency and seemed to reflect the more polymerised nature of the OM in the ACT-IND sludge.

Those from P2 showed the multi-coloured black, red, yellow, and orange hues associated with the oxidation of Fe^{2+} and the precipitation of ferric hydroxides typically observed in plinthic soils. These observations seem to reflect the fluctuating redox conditions resulting from anaerobic digestion followed by exposure to oxygen when dried and later composted.

Due to the drier nature of the DIG-MUN sludge, the aggregates or "clots" found in P2 did not occur in association with an outer layer of sawdust as occurred with the dewatered sludge. Those from P3 and P4 were slightly more grey-brown in colour (as opposed to the brown colour of the ACT-MUN sludge in these piles) but apart from that, appeared to be of the same consistency.

The bottom right picture of a piece of wood chip from one of the piles seemed to be a rather nice display of humification occurring from the outer edges inwardly.

72
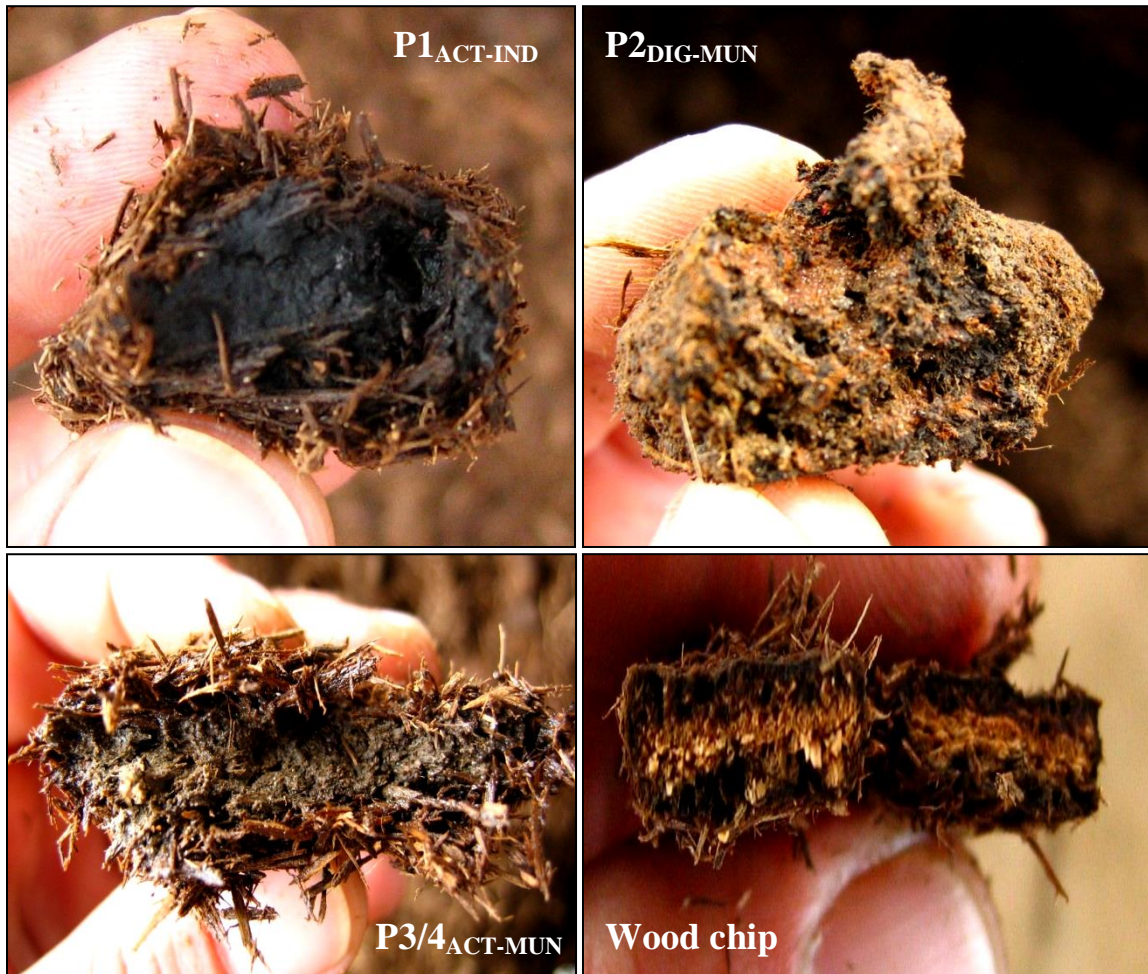
Figure 3.18 "Sludge aggregates" after 139 days of composting and humification observable from the outer edges of a wood chip

3.4   Summary

From a composting process point of view, it is clear that dewatered ACT-IND and ACT-MUN sludge can be successfully composted with a carbonaceous bulking agent like sawdust when established composting principles are applied. DIG-MUN sludge that has undergone evaporative drying on the other hand did not produce a satisfactory composting mixture as evidenced by the measured process parameters for P2 (temperature profile and decomposition deducted from change in volume). This can be ascribed to a low pH (4.88) possibly inhibiting microbial activity and to the substantially lower C and N content as well as higher ash content of P2 compared to the other three

73

composting mixtures. These feedstock characteristics are the direct result of the characteristics of the DIG-MUN sludge - indicating that it is already sustantially stabilised (and mineralised) and contains insufficient biodegradable organic matter (and therefore insufficient energy) for effective composting. Therefore, little benefit is received from the composting of the $WWS_{DIG-MUN}$ with sawdust.

Suitable composting mixtures can be formulated with reasonable accuracy and the preferred range of conditions for composting can be achieved from a limited number of known raw material characteristics – most importantly water, C and N content. The high water content of dewatered sludge seems to be the limiting factor determining the maximum sludge to sawdust ratio that can be successfully composted. For the particular dewatered sludge used in this experiment sludge-to-sawdust ratio (volume) of about 1:6 or even 1:7 would be recommended for ACT-IND sludge and about 1:3 for ACT-MUN sludge. Some variation can be expected between different batches of materials and also due to material handling and volumetric measurement methods.

Piles of 4 m^3 in size were fairly vulnerable to cold wind and the low ambient temperatures during winter months. Large scale windrows or big static piles should be much more buffered against environmental factors and therefore achieve more sustained high composting temperatures and more "typical" temperature profiles in suitable mixtures. Mechanical windrow turners are expected to largely eliminate the "clumping" observed with manual turning. The covering of piles with plastic sheeting was absolutely necessary for the successful completion of the experiment but did influence the natural draught ventilation and water dynamics of piles.

The physical parameters employed to monitor composting i.e. temperature, water content and pile volume, together with the human eye and nose (to observe dry or anaerobic conditions), were found to provide adequate information about the dynamics of the process for timely management responses like water addition and turning.

Although composting can be monitored more closely and accurately in better controlled experimental conditions like in reactor studies, designing the experiment with a practical composting situation in mind allowed for valuable observations and experiences of the impact that changing and seasonal environmental conditions have on the process.

3.5　References

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Chapter 4: The evolution of certain chemical and biochemical parameters during composting and final compost quality characteristics

4.1 Introduction

The temporal changes in certain chemical and biochemical characteristics observed in composting materials, give insight into the basic processes that constitute composting. These observations also provide insightful parameters for the evaluation of the suitability of raw materials and feedstock for composting, systematic monitoring as well the evaluation of compost quality. Furthermore, compost produced from different WWS and Eucalyptus sawdust (bulking agent) can be expected to be of a different quality.

This chapter aims to present and interpret the changes in chemical and biochemical parameters observed during the experimental composting of three different types of WWS and Eucalyptus sawdust as described in Chapter 3. The objectives are to:

- Obtain a data set of selected chemical and biochemical parameters over the period of composting with special emphasis on the state of these parameters at the start of composting (essentially the initial characteristics of mixtures), at the end of active (thermophilic) composting and at the end of curing (final product).
- Interpret this data set in terms of the physical parameters measured during composting and the microbial processes giving rise to characteristic chemical and biochemical changes as described in literature.
- Evaluate the end product quality of treatments and compare it to chosen benchmarks.

4.2 Materials and methods

Samples from the four compost treatments discussed in Chapter 3 were analysed to determine the chemical and biochemical transformations that occurred in the materials during composting and the final quality characteristics of the compost produced. In the determination of final quality characteristics, two reference composts namely a

77

commercially available compost that does not contain any WWS ($C1_{NO\ WWS}$) and a commercially available sludge based compost ($C2_{WWS}$) were also analysed.

4.2.1 Temporal chemical and biochemical changes during composting

Laboratory analyses of material samples (3 replicates) consisted of the following:

- Total C and N content were determined with a Carlo Erba NA 1500 C/N/S Analyzer (see Appendix A for detail).

- Potentiometric pH was measured in a 1:10 (mass base) water extract after equilibrating for 24 hours (stirred initially when mixed and before pH was measured) using a pH electrode and pH meter calibrated against buffers at pH 4 and pH 7.

- Electrical conductivity (EC) was measured with a suitable conductivity electrode and meter in the same water extract as pH.

- Ash/volatile solids content was measured gravimetrically after dry combustion of oven dry samples at 550°C for 4 hours in a muffle furnace (Thompson *et al*., 2002).

- Acid Detergent Fibre (ADF), Acid Detergent Lignin (ADL) and Neutral Detergent Fibre (NDF) were determined through the methods described by Goering and van Soest, 1970 (analysed by Nutrilab, University of Pretoria). Organic fractions were calculated as follows:

    - Cellulose (% of dry matter) = ADF (% of dry matter) – ADL (% of dry matter);

    - Hemi-cellulose (% of dry matter) = NDF (% of dry matter) – ADF (% of dry matter);

    - Lignin (% of dry matter) = ADL (% of dry matter); and

    - Soluble organic matter (% of dry matter) = 100% - (Cellulose + Hemi-cellulose + Lignin + Ash).

- NaOH extractable carbon (humic-like substances) was determined by combining a 4g compost sample with 40 cm^3 1 M NaOH solution and shaking on a reciprocal shaker for 24 hours. The samples were centrifuged, the liquid decanted and the extraction repeated with 20 cm^3 1 M NaOH solution – again shaken for 24 hours, centrifuged and decanted. The total decant from extraction step one and two was vacuum filtered (Thompson *et al*., 2002).

- The quantity of organic carbon in the filtrate was determined through the Walkley Black method for determining organic carbon in soil (The Non-Affiliated Soil Analysis Work Committee, 1990) modified by using the 10 cm^3 NaOH extract instead of 1g soil.

4.2.2 Final compost quality characteristics

Laboratory analyses of samples (3 replicates) consisted of the following:

- Water holding capacity and air filled porosity were determined by filling a cylinder of known diameter with moist compost material and using a standard weight applied for a standard time to compact the sample. The final height (and final volume) of the sample was determined and the compacted sample slowly saturated with water to displace all trapped air and soaked for 30 minutes. The saturated sample was allowed to drain freely for 30 minutes and the volume of drained water as a percentage ratio of the sample volume, represents the air filled porosity at maximum water holding capacity (field capacity). The sample was oven dried (75º) and the mass (volume) of water so determined as a percentage ratio of the total sample volume represents the water holding capacity. The sum of the air filled porosity and the water holding capacity represents the total porosity (Handreck and Black, 1999).
- Phytotoxicity bioassay: Seedling emergence and relative growth (vigour) in potting media was determined through direct seeding using distilled water as the moisture source (see Appendix A for detail).
- Cation Exchange Capacity (CEC) was determined through the displacement of cations on exchange sites in the sample with NH_4^+ cations by leaching with an ammonium acetate buffer solution (see Appendix A for detail).
- Elemental analysis involved:
  - A perchloric/nitric acid sample digestion wherein 1g of sample is digested with 7ml HNO_3 (conc. nitric acid) and 3ml $HClO_4$ (perchloric acid) at temperatures up to 200ºC and brought to volume in a 100ml volumetric flask; and
  - ICP determination of Ca, Mg, P, K, Na, Fe, Zn, Mn and Cu (see Appendix A for detail).

79

Statistical analysis of data was performed with SAS 9.1 and Microsoft EXCEL.

4.3 Results and discussion

4.3.1 Temporal chemical and biochemical changes during composting

N transformations during composting have a marked effect on the pH and EC of composting materials (Sanchez-Monedero *et al.*, 2001) and supply information on certain microbial processes that occur during composting.

In Figure 4.1 (pH) and Figure 4.2 (EC) the data series P1, P2, P3 and P4 represent pH/EC data for oven dried samples that were primarily taken to determine water content at a number of time intervals during composting.
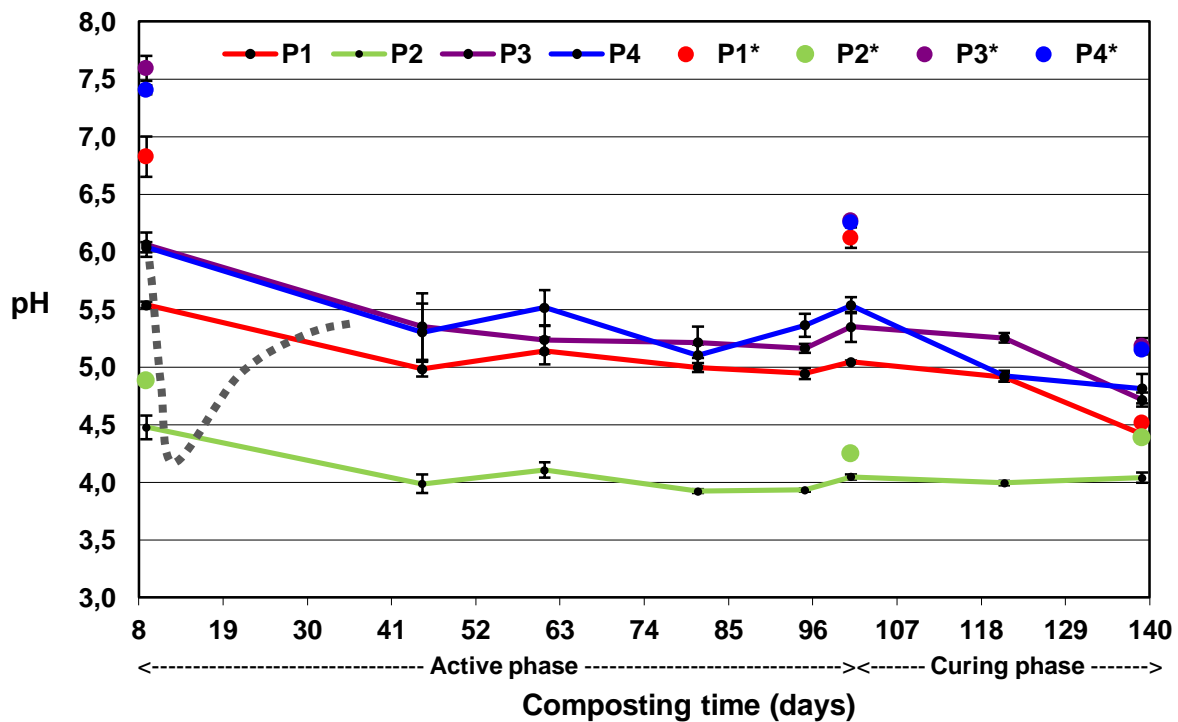
Figure 4.1 Change in pH of compost piles over 139 days of composting (error bars represent standard deviations - three replicates, ▪ ▪ ▪ ▪ expected pH behaviour over the initial stages of high rate thermophilic composting)

The data series P1*, P2*, P3* and P4* represent pH/EC determinations on samples without any drying (as sampled) with its water content taken into account in preparation of the 1:10 (mass base) extract. These samples were taken shortly after the start of composting (day 9, after visually satisfactory initial mixing were achieved), at the end of the active/thermophilic phase (day 101) and at the end of the curing phase (day 139). It was so taken to measure the transformation of certain parameters over the composting period with specific comparison of values at the start of composting, after the active phase of composting and after the curing phase of composting.

Composting starts at ambient temperature with microorganisms decomposing the easily degradable carbon sources like monosaccharide, starch and lipids. Because organic acids result from this, the pH initially decreases (Crawford, 1983). The pH can also initially decrease due to increased carbonation from elevated CO_2 levels in compost piles (Haug, 1993).

Thereafter, microorganisms start to degrade proteins and the resultant mineralisation of organic N yields ammonia which hydrolyses to form ammonium, a proton (H^+) consuming reaction that contribute to an increase in pH, especially during the initial stages of high rate thermophilic decomposition (Haug, 1993).

$$R\text{-}NH_2 + 2H_2O \leftrightarrows OH^- + R\text{-}OH + NH_4^+$$
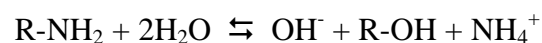
The extent to which this effect is observed depends on substrate degradability (usually linked to lignin/lignocellulose content and indices), pH and intensity of microbial activity. Microbial activity is influenced by environmental conditions like pH, oxygen supply, water content and temperature. The uptake of reduced N by microorganisms in all likelihood involves the exchange of a proton (H^+) into the solution when NH_4^+ (or other cations) is taken up - resulting in an increase in pH.

At pH above 7.5 (not observed in any of the experimental piles) substantial N losses can occur through ammonia volatilisation which will increase with increasing substrate

degradability (low contents of lignin/lignocellulose), microbial activity, aeration and increasing pH (Haug, 1993). Ammonia volatilisation constitutes the removal of reduced N from the system (solution). This reduces the potential for future acidification (from the oxidation and uptake of reduced N) and therefore potentially increases the pH.

$$NH_4^+{}_{(aq)} + OH^-{}_{(aq)} \leftrightarrows H_2O_{(l)} + NH_{3(g)}$$
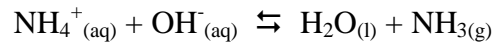
The neutralisation reaction of the added lime would also have raised the pH of compost piles.

The pH data of P1, P3 and P4 do not show these expected trends during the initial stages of high rate thermophilic composting due to a lack of short term data. The dashed line in Figure 4.1 represents the "expected" pH profile in the light of the processes discussed above and puts the net decrease in pH of P1, P3 and P4 as shown by the data, in perspective.

As composting proceeds, organic acids are further decomposed and CO_2 levels drop as microbial activity slows and less degradable organic matter remain. These opposing changes result in the typical buffering of pH observed over the middle and latter stages of the active phase of composting (Cundiff and Mankin, 2003 and Wong and Fang, 2000).

After the easily degradable carbon sources have been depleted, the more resistant compounds such as cellulose, hemicellulose and lignin are degraded and partly transformed into humus (Crawford, 1983).

The data in Figure 4.1 reflect these processes over the period from day 45 to day 101 (end of active phase) when pH values of piles were well buffered after the decline from initial feedstock values to end the active phase at values (as sampled, pH) of 6.12 (P1*), 6.27 (P3*), 6.26 (P4*).

At the onset of the curing phase when the temperature falls below 40° C, nitrification sets in and the conversion of ammonium to nitrate results in a more pronounced decrease in pH (Sanchez-Monedero *et al.*, 2001) due to the formation of two protons (H^+).

$$NH_4^+ + 1½\ O_2 \leftrightarrows NO_2^- + 2H^+ + H_2O + energy$$
$$NO_2^- + ½\ O_2 \leftrightarrows NO_3^- + energy$$
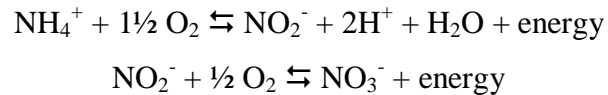
As long as conditions are favourable for both reactions, the second reaction follows closely after the first to prevent the accumulation of nitrite. These conditions include enough oxygen for the oxidation reactions to occur, suitable water content and an abundance of exchangeable base-forming cations like Ca^{2+} and Mg^{2+}, rather than pH itself (Brady & Weil, 1999). The extent of nitrification will further depend on the amount of ammonium present and usually continues up to the end of the curing phase (Sanchez-Monedero *et al.*, 2001).

Over the 38 day curing phase the pH of fresh samples declined more sharply from day 101 values to final values (end of curing phase, as sampled, pH) of 4.52 (P1*), 5.18 (P3*) and 5.15 (P4*) indicating that nitrification was indeed prominent during this phase of composting.

The nitrogen in the stabilised WWS_{DIG} in P2 was, however, already largely mineralised (+/- 50% of total N) and nitrified (+/- 50% of mineral N) and microbial decomposition was far less pronounced in this pile. With its temperature mostly remaining below 40° C, the pile never exhibited a true thermophilic phase and the decline in the already low initial pH of P2* (4.88) was probably caused by early nitrification during the first 45 days of composting. Thereafter, the pH was well buffered and largely remained so to the end of composting (pH of P2* of 4.39). This indicates that most of the ammonium in P2 was already converted to nitrate by the end of day 45.

The pH values of oven dried samples (P1, P2, P3 and P4) were consistently lower than that of fresh samples (P1*, P2*, P3* and P4*), indicating that oven drying (100 ºC, 24

83

hours) leads to an increase in the oxidation of reduced compounds including organic C and N that tends to decrease the pH. This effect was less pronounced in P2 due to its lower C (and N) content. Initial pH values of feedstock (as sampled) and reasons for differences were discussed in detail in Chapter 3, p. 59.

The EC of composting materials is influenced by a net increase or decrease in ion concentration and, therefore, dominance of either N mineralisation (ion forming) or N immobilisation (ion consuming) by microorganisms, throughout the composting period.

The EC profiles of P1, P3 and P4 (Figure 4.2) remained flat over the first 45 days of composting despite all evidence that prolific mineralisation was occurring. This is a clear indication that most of the ammonium (and nitrate) resulting from mineralisation, was assimilated by the growing microbial population and the processes of N mineralisation and N immobilisation were in equilibrium.
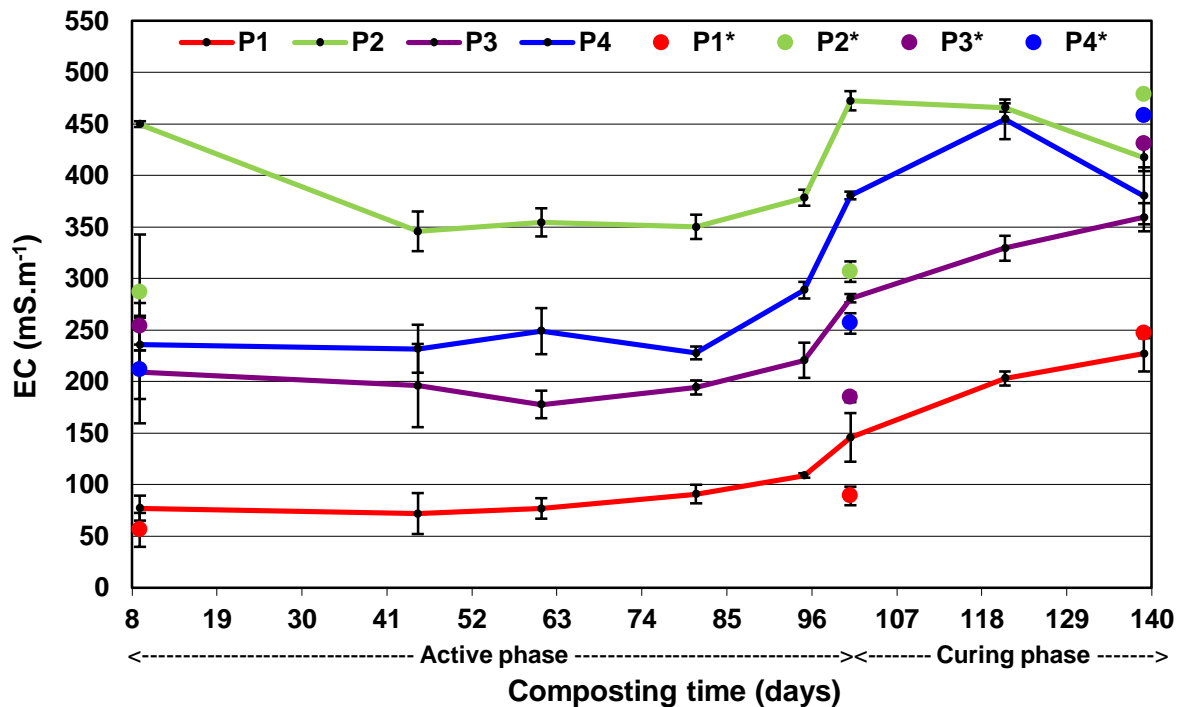
Figure 4.2 Change in EC of compost piles over 139 days of composting (error bars represent standard deviations - three replicates)

Only once the reasonably degradable substrates were depleted towards the end of active composting, a collapse and the resultant mineralisation of the thermophilic microbial population occurred. This resulted N mineralisation starting to dominate over immobilisation and the EC started to increase due to an increase in free ammonium. The EC of piles at the end of the active phase (as sampled, $mS.m^{-1}$) of 88.7 (P1*), 185 (P3*), and 256 (P4*) were marginally higher than initial feedstock values (except for a decrease in EC for P3* which is not supported by the P3 dataset and is therefore inconclusive).

After the onset of the curing phase, the combined processes of re-mineralisation and nitrification result in a continued increase in EC to the end of composting.

The decline in EC of P2 over the first 45 days of composting indicates a net decrease in ion concentration due to inorganic N assimilation being dominant over mineralisation, depleting available N and causing a period of nitrogen draw down (N-negative period). This was coupled with the low C, N and organic N content of P2 and the high level of stability of the digested sludge. Temperature, pH and the fact that a substantial amount of NH_4^+ was present, support the fact that nitrification was occurring simultaneously.

Thereafter, the EC of P2 remained flat from day 45 onwards and then started to increase over the period from day 81 to day 101 due to net N mineralisation from microbial decline to end the active phase on 307 $mS.m^{-1}$ (P2*). During the 38 days of curing, the EC in fresh samples of P2 continued to increase towards the end of composting while the EC measured in oven dried samples decreased, making it inconclusive whether N mineralisation or N assimilation was dominant.

The pH and EC data for P1 and P3 reflect nitrification and net N mineralisation occurring during and up to the end of the curing phase (decreasing pH and increasing EC). The pH of P4 dropped more pronouncedly over the initial part of curing (day 101 to day 121) and then still declined from day 121 to day 139 but at a slower rate indicating that nitrification had slowed down. This can occur when most of the ammonium in P4 was converted to nitrate by day 121. The EC data for P4 (increasing up to day 121 and then

85

declining towards the end of curing) indicate a switch over from net mineralisation to net assimilation of N that can occur after a sudden slowdown in microbial activity followed by re-growth of the population. This could be an indication that there remained degradable substrates in P4 and that composting may have not been complete for this treatment.

Ash content (Figure 4.3) is a measure of the mineral component of total solids (that does not volatilise when combusted at 550°C for 4 hours) that can be assumed to be conservative i.e. the total mass of ash in compost piles does not markedly change over the composting period.
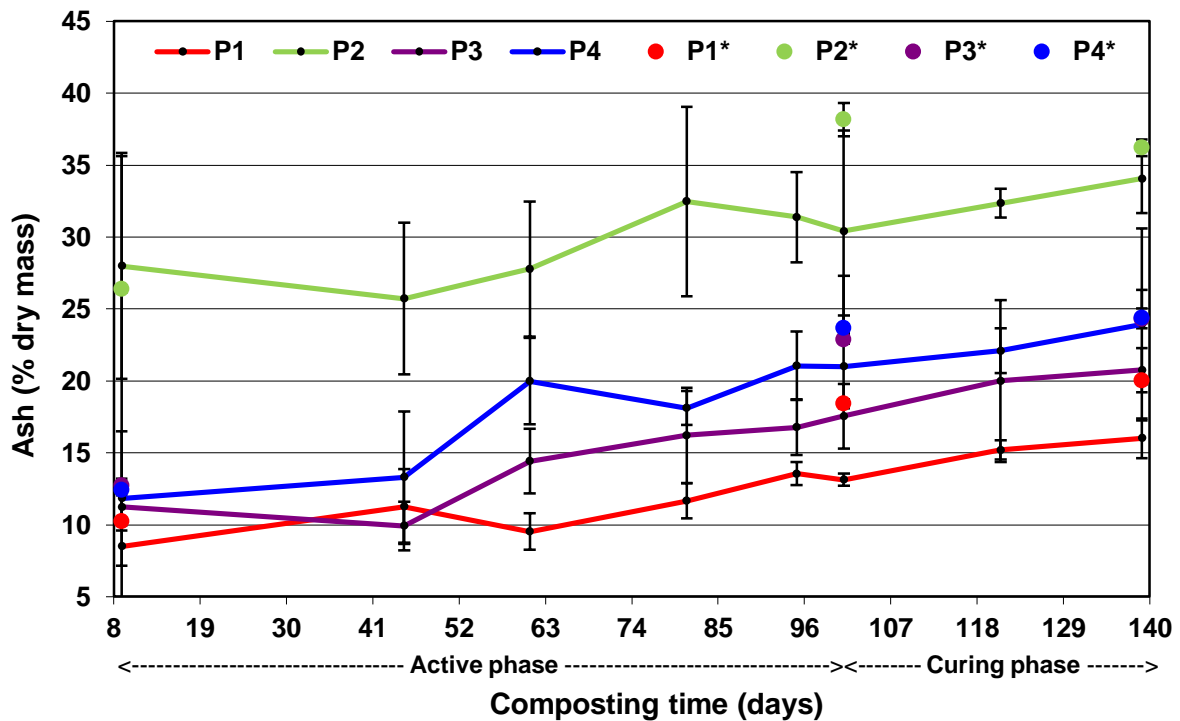
Figure 4.3 Change in ash content of compost piles over 139 days of composting (error bars represent standard deviations - three replicates)

The observed increase in ash concentration over the composting period is the result of a decrease in its reciprocal (100 − ash content, %) which is a measure of the organic component of total solids referred to as volatile solids (VS) or loss on ignition organic matter (LOIOM) (Figure 4.4). The biodegradable volatile solids (BVS) component of VS

86

supplies the energy for the microbial decomposition of composting material and is expected to decrease during composting (Thompson *et al.*, 2002).

In Figure 4.3 (ash) and Figure 4.4 (VS) the data series P1, P2, P3 and P4 represent ash/VS data for un-milled oven dried samples and the series P1*, P2*, P3* and P4* represent ash/VS determinations on samples that was milled before oven drying. These samples were taken on day 9, day 101 (end of the active/thermophilic phase) and on day 139 (end of the curing phase).
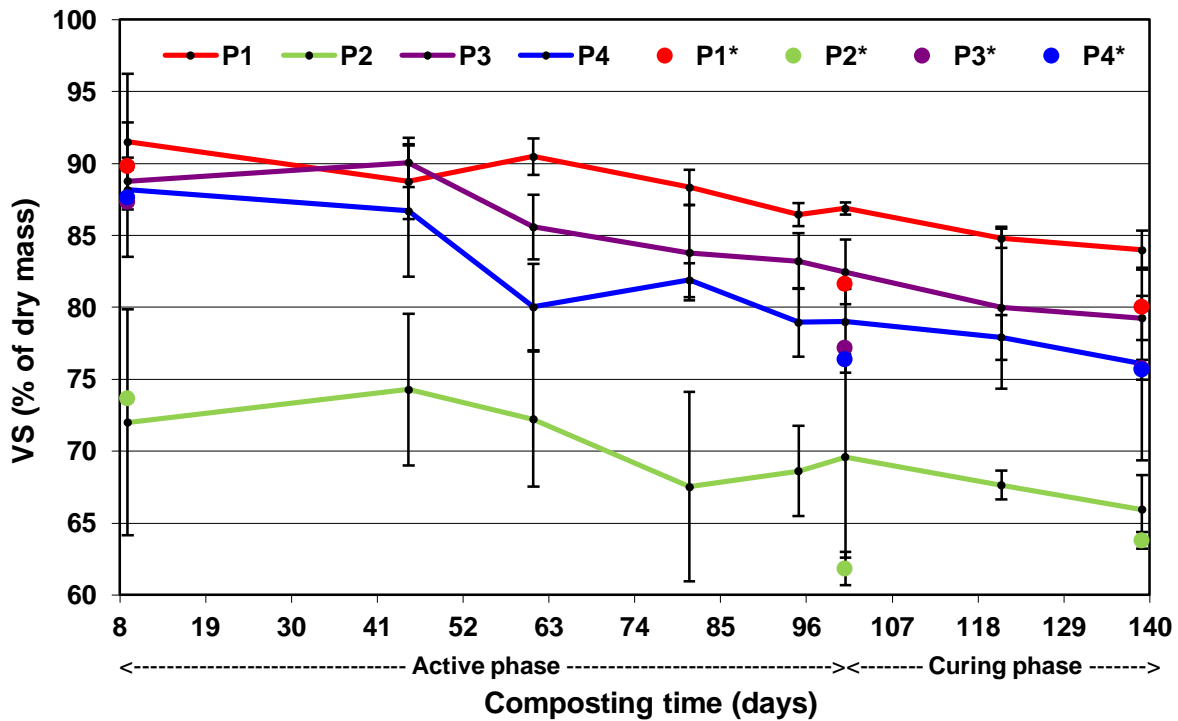
Figure 4.4 Change in volatile solids content of compost piles over 139 days of composting (error bars represent standard deviations - three replicates)

The milled samples mostly produced ash values that were higher (and VS values that were lower) than those of un-milled samples. The effect could be due to less effective drying of aggregates in un-milled samples possibly accompanied by less volatilisation, overstating dry mass and therefore understating ash content. It could also be due to more effective oxidation of the homogenised (milled) samples during combustion. The value

determined for P2* (milled sample) on day 101 looks like an "outlier" if compared to the P1 (un-milled sample) profile.

The VS content of compost piles showed a steady decreasing trend through the entire composting period. Considering the data for milled samples (P1* etc.), the rate of decrease in VS content was higher during the 101 days of thermophilic temperatures than during the curing phase. This "decomposition rate" represents only the relative decomposition of VS or LOIOM through the decrease in dry mass concentration and a more absolute rate of decomposition is obtained when ash content is assumed to be conservative (i.e. constant mass).

The conservation of ash assumption leads to the following equations (example for P1):

$OM_{decomposed}$ on day 101 is calculated as follows: $100 - (Ash_{day9}/Ash_{day101} \times 100)$; and

$OM_{remaining}$ on day 101 is calculated as follows: $VS_{day101}/100 \times (Ash_{day9}/Ash_{day101} \times 100)$.

Table 4.1 contains the data set of measured ash content and calculated organic matter values.

Table 4.1 Organic matter decomposition in P1$_{ACT-IND}$ as calculated from measured ash content and assuming ash conservation

Day	Ash$_{milled}$	VS$_{milled}$	Ash$_{conservative}$	OM$_{decomposed}$	OM$_{remaining}$	Total Phases
	(%) dry mass					
9	10.2	89.8	10.2	0.00	89.8	100
101	18.4	81.6	10.2	44.4	45.3	100
139	20.0	80.0	10.2	48.9	40.9	100

Figure 4.5 depicts this absolute decrease in VS or LOIOM for P1$_{ACT-IND}$ (green shaded area, shown as an example) through decomposition. The average rate of decomposition

88

that is represented by the gradient in Figure 4.5 decreased from 0.5% daily during the active phase, to 0.2% daily during the curing phase. This point to the general notion that although the decomposition of certain organics still continue during the curing phase, the rate of decomposition drastically declines and biochemical processes/transformations like humification become more dominant in this phase (Cundiff & Mankin, 2003).
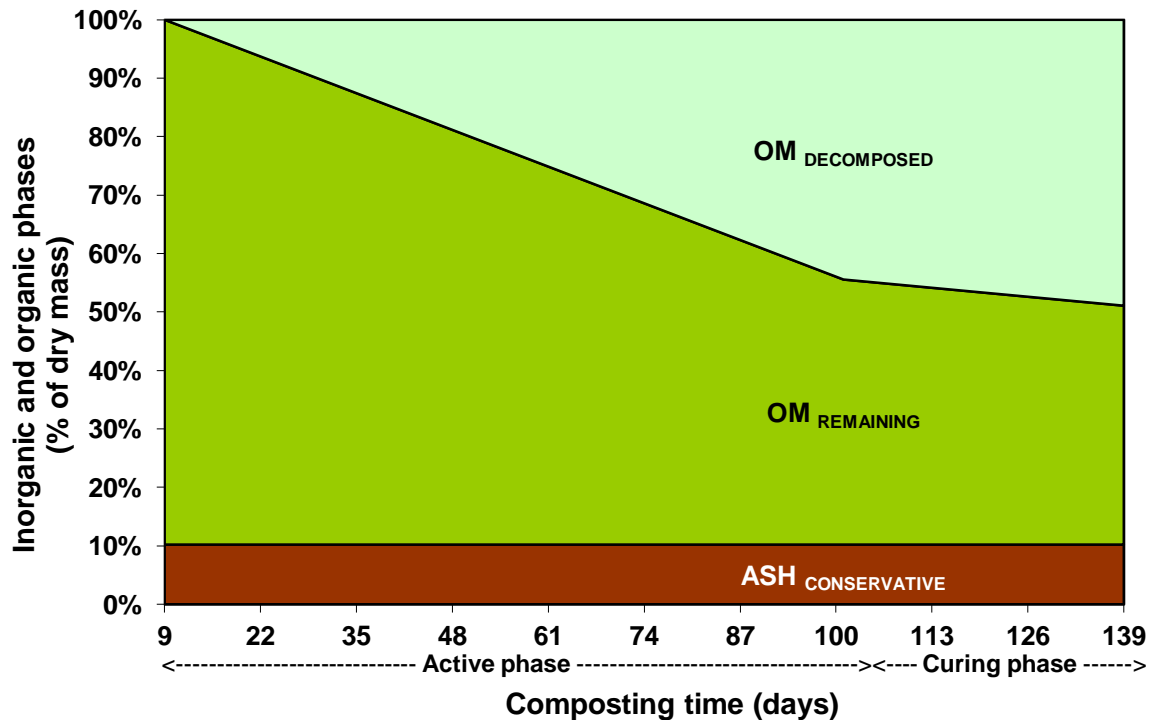
Figure 4.5 Organic matter decomposition in P1$_{ACT-IND}$ over 139 days of composting and specifically over the active and curing phases (assumed: conservation of ash)

The decomposing microorganisms utilise the C source (organic matter) mainly for energy and only a small fraction is incorporated into the microbial biomass (C:N ratio $\approx$ 8). The energy from the oxidation of C that is not utilised for microbial metabolism is released as heat and CO_2 is lost from the system (Tuomela *et al*, 2000).

The total C content of piles (Figure 4.6) at any given time after the start of composting represents the net of C lost as CO_2 and the concentration of C in the remaining material due to mass loss. The total C content decreased for P1 (1.56%), P3 (7.71%) and P4 (1.42%) over the entire composting period. This indicates that C loss was greater than C

89

concentration (which was substantial with ≈ 50% reduction in pile volume) and could be seen as evidence of high rate decomposition.

Contradictory to this, the total C content of P2 increased over the entire composting period by 3.6%. This indicates that C concentration (which was far less substantial with only 17% volume reduction) was greater than C loss and therefore supplies evidence of the low rate of decomposition illustrated by its temperature profile and volume reduction. P2 had a low initial C content of 24.0% and low C:N ratio of 19.3 (Figure 4.9).
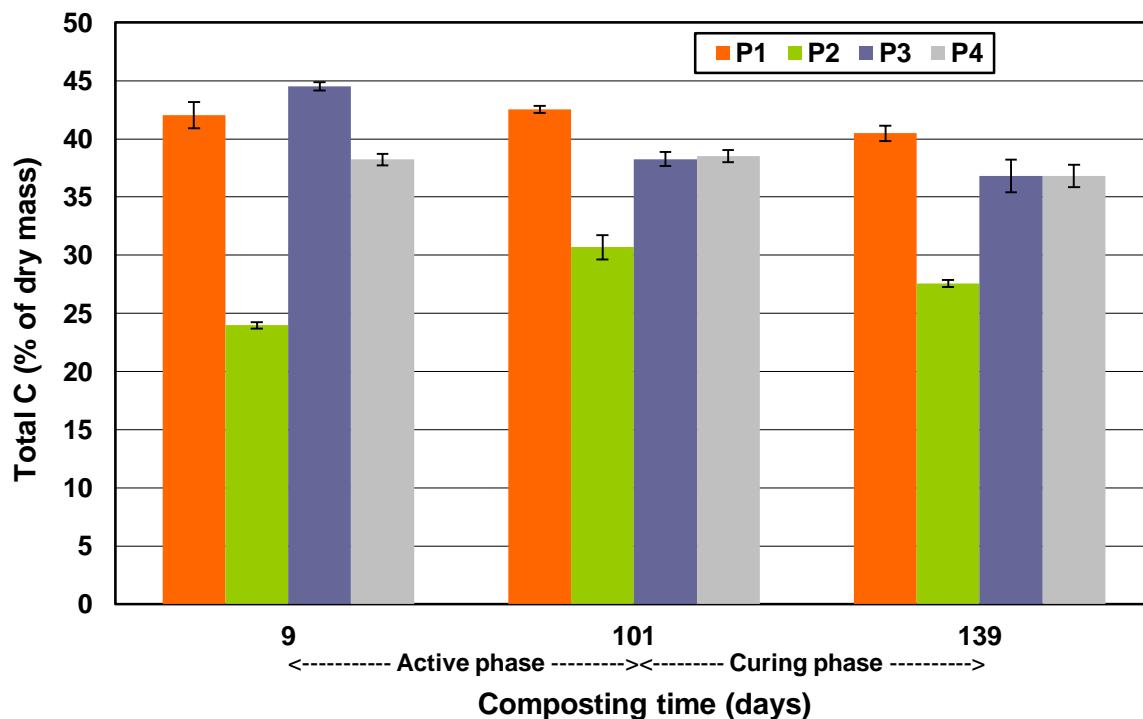
Figure 4.6 Change in total C content of compost piles over 139 days of composting and specifically after the active and curing phases (error bars represent standard deviations – three replicates)

The relatively high decrease in C content of P3 of which most occurred over the active phase (6.26%) is coupled with a high initial C content (44.5%) and high C:N ratio (30.9). P1 similarly had a high initial C content (42.0%) and high C:N ratio (29.7) but its C content remained steady over the active phase and only marginally decreased (1.56%) over the entire composting period. Although the C:N ratio of P1 were within the

90

acceptable range (be it on the high side), the actual amount of sludge (681 kg) added to the 4 m^3 of sawdust (and because of the formation of sludge aggregates (Chapter 3), were too little to achieve a good distribution (of N) throughout the pile. Therefore, N deficiency in large areas in the pile can be regarded as the main reason for inhibited decomposition of especially sawdust C in this pile.

The 1.42% decrease in total C content of P4 over the composting period is coupled with a relatively high initial C content (38.2%) and a low C:N ratio (20.3), yet C decomposition was as stifled as in the case of P1 (judged by the net change in total C content). With little chance of N deficiency, the only possible reason must be the high initial water content or more specifically the large(r) amount of sludge aggregates distributed through the pile that compromised its porosity and possibly inhibited C decomposition to some extent.

The alkali extractable carbon to total organic carbon ratio is often used as a humification index to evaluate the degree of humification and compost maturity. If one considers the NaOH extractable C fraction (here expressed as a percentage of total C) of the piles (Figure 4.7) the data shows a consistent increase in the "pool of humic like substances" over the active phase for P1, P3 and P4.

This trend can be expected to occur over the thermophilic phase of composting when more resistant organic compounds like cellulose, hemi-cellulose and lignin are degraded. The decomposition products from these compounds (especially lignin) are seen to be important pre-cursors (humic like substances) for the process of humification. The relatively large increase in the NaOH extractable C fraction of P1 (9.37% compared to 4.87% and 3.26% for P3 and P4 respectively) over the active phase of composting may be the result of the industrial origin of the ACT$_{IND}$ sludge and its chemical composition (verbally reported to be highly aromatic).

From Chapter 2, Tuomela *et al.* (2000) report that immature compost generally contains high levels of fulvic acids and low levels of humic acids. The humic acid fraction increases as decomposition proceeds while the fulvic acid fraction either decreases or

remains unchanged. Aliphatic material is lost by mineralization while enrichment in aromatic structures occurs (Garcia *et al.*, 1992).
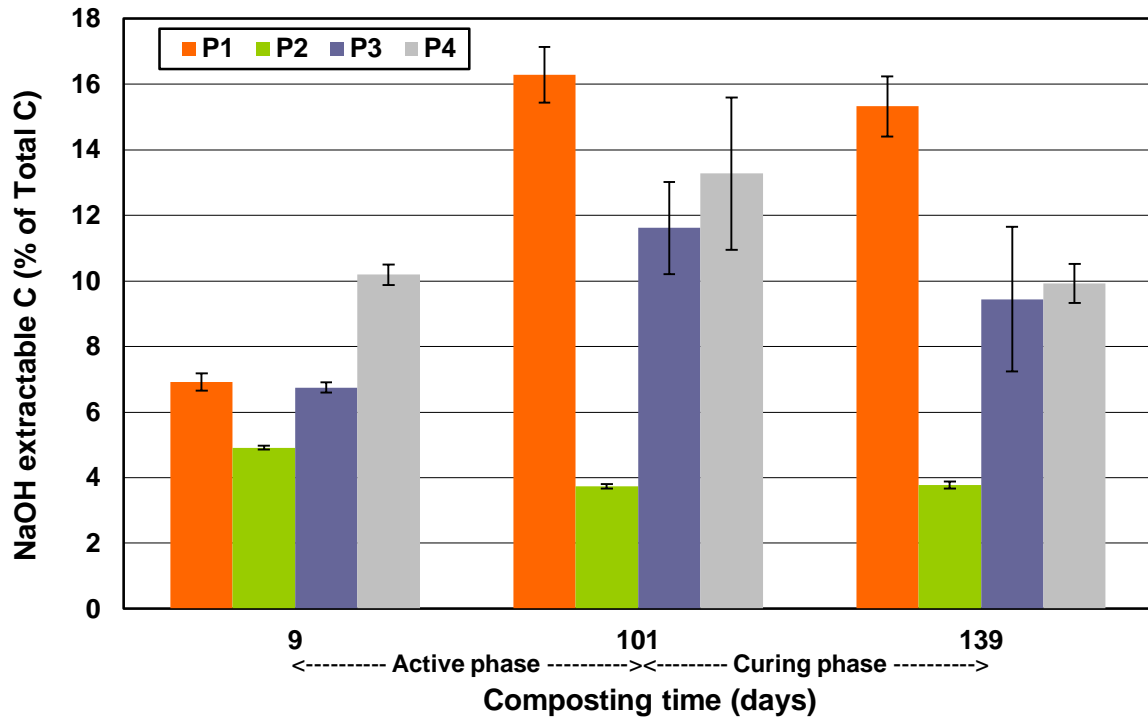
Figure 4.7 Change in NaOH extractable C (humic-like substances) of compost piles over 139 days of composting and specifically after the active and curing phases (error bars represent standard deviations - three replicates)

The NaOH extractable C pool in P1, P2 and P3 decreased over the curing phase. This trend supports the abovementioned observations that fulvic and later humic acids are synthesised from the pool of humic like substances. This, together with the mineralisation of aliphatic and enrichment in aromatic structures, resulted in the observed decrease in the mass concentration of NaOH extractable C over the curing phase, supporting another general notion that it is over this phase of composting that humification mainly occurs.

In the case of P2, and supported by the temperature data from Chapter 3, the evolution of NaOH extractable C indicate that the pile was in a curing phase from the start. It's comparatively low content of humic-like substances decreased over the active phase and remained relatively constant over the curing phase. This trend indicates that humification

92

occurred from the start of composting and that the low rate of mineralisation in this pile, resulting in few decomposition products being added to the humic like pool, caused a complete slowdown in humification during the latter part of composting.

N consistently accumulated in all compost piles over the composting period and mostly so over the active phase of composting (Figure 4.8). Nitrogen is a critical component of the proteins, nucleic acids, amino acids, enzymes and co-enzymes necessary for microbial cell growth and functioning. The degradation process will be slow if nitrogen is a limiting factor. Excess nitrogen, on the other hand, is often lost from the system as ammonia gas or other nitrogen compounds (Golueke, 1991).

Figure 4.8 Change in total N content of compost piles over 139 days of composting and specifically after the active and curing phases (error bars represent standard deviations - three replicates)
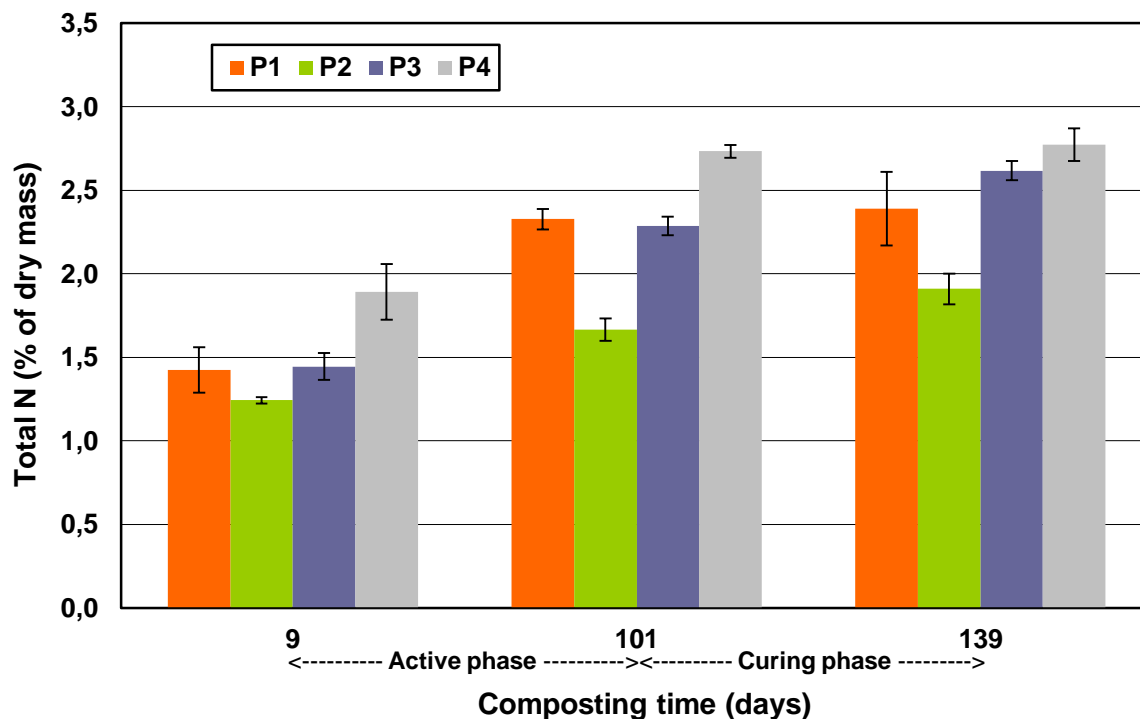
The observed increase in N content is an example of the concentrating effect that occurs in aerobically composting material due to mass loss. It can be assumed that little or no N was lost through ammonia volatilisation as the measured pH values of compost piles

93

were below pH 7.5 at all times (except for the initial pH of P3). Therefore, disregarding other possible gaseous N losses (de-nitrification), one can assume that most of the initial N concentrated in the composting material as pile dry mass decreased through OM decomposition and the resultant evolution of CO_2 and H_2O.

Resultant from the change in C and N content, the C:N ratios of compost piles decreased over the composting period to values around 15 (Figure 4.9). The optimum C/N ratio has been reported to be 25 to 40, but the value varies depending on the substrate (Golueke, 1991). According to this, the initial C:N ratios of P2 and P3 were below optimal but the only real risk to this is that substantial N losses can occur. The C:N ratios of all piles decreased into a fairly narrow range between 17.05 (P1) and 13.29 (P4), which is typical for compost (Brady and Weil, 1999) and again reflecting the processes of degradation and humification.
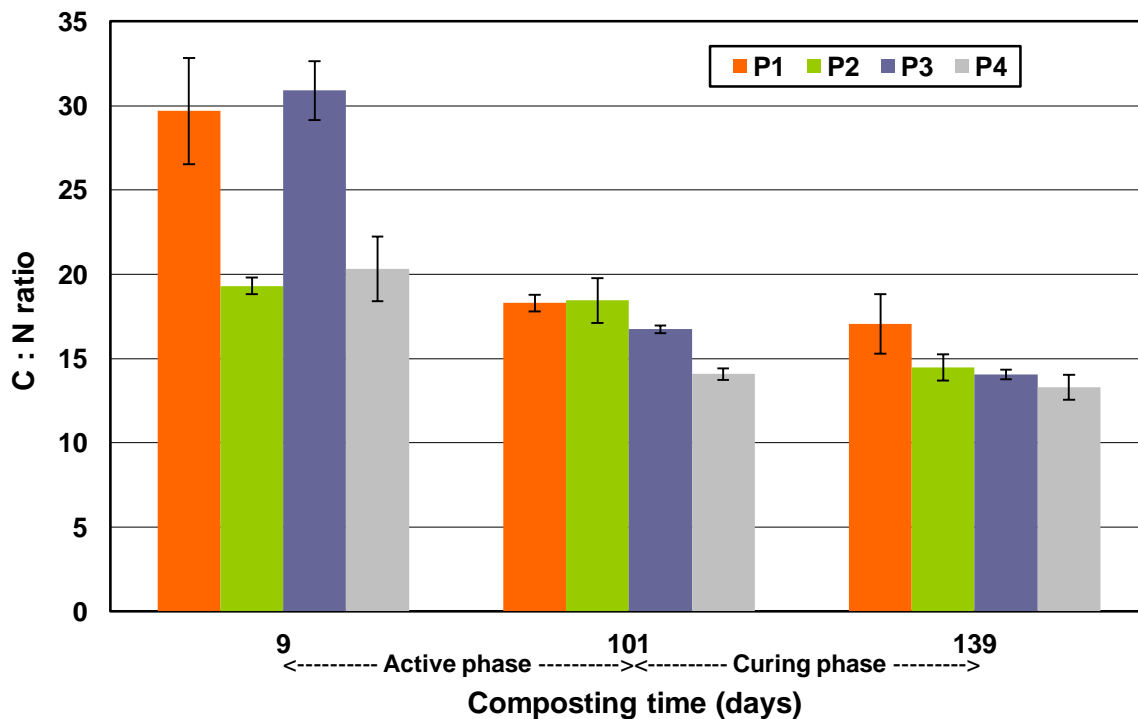

Figure 4.9 Change in C:N ratio of compost piles over 139 days of composting and specifically after the active and curing phases (error bars represent standard deviations - three replicates)

94

Biochemical characterisation of composting materials was performed through the fractionation of samples by means of the Van Soest method (Table 4.2). The lignin content (% of dry mass) increased in all compost piles over the active phase and slightly increased or slightly decreased over the curing phase of composting.

Table 4.2 Change in concentration/content of organic matter constituents of compost piles as calculated from measured fibre fractionation by acid and neutral solvents according to the "Van Soest" method over the two phases of composting (± standard deviation – three replicates)

PILE	DAY	ADF (%) dry mass	NDF (%) dry mass	ADL/LIGNIN (%) dry mass	$CELL_{calc}$ (%) dry mass	$HCELL_{calc}$ (%) dry mass	SOL_{calc} (%) dry mass
P1$_{ACT-IND}$	9	73.5 ±0.53	81.1 ±0.88	18.6 ±0.38	54.9 ±0.51	7.63 ±0.88	8.69 ±0.69
P1$_{ACT-IND}$	101	64.0 ±1.75	68.4 ±0.99	30.9 ±0.73	33.1 ±1.39	4.43 ±2.16	13.2 ±0.95
P1$_{ACT-IND}$	139	62.1 ±1.59	59.8 ±0.97	29.7 ±0.55	32.4 ±2.10	0.00 ±0.00	17.9 ±1.72
P2$_{DIG-MUN}$	9	54.1 ±1.21	55.2 ±0.44	14.2 ±0.35	39.9 ±0.87	1.18 ±1.37	18.4 ±0.39
P2$_{DIG-MUN}$	101	43.8 ±1.20	42.1 ±0.64	15.8 ±0.30	27.9 ±1.17	0.00 ±0.00	18.1 ±0.57
P2$_{DIG-MUN}$	139	45.9 ±0.87	42.4 ±1.16	14.9 ±0.38	30.9 ±0.92	0.00 ±0.00	17.9 ±0.88
P3$_{ACT-MUN}$	9	71.0 ±0.84	77.2 ±0.37	18.6 ±0.44	52.4 ±0.70	6.18 ±0.81	10.1 ±0.21
P3$_{ACT-MUN}$	101	62.6 ±0.98	65.3 ±0.88	24.5 ±0.60	38.0 ±0.85	2.72 ±1.76	11.9 ±0.67
P3$_{ACT-MUN}$	139	57.1 ±0.87	58.9 ±1.06	25.7 ±0.55	31.4 ±1.40	1.83 ±0.92	16.9 ±0.89
P4$_{ACT-MUN}$	9	64.4 ±1.19	72.4 ±0.44	18.1 ±0.47	46.4 ±0.75	7.99 ±1.23	15.2 ±0.48
P4$_{ACT-MUN}$	101	54.3 ±0.96	56.7 ±1.27	23.1 ±0.23	31.2 ±0.74	2.39 ±0.71	19.7 ±2.02
P4$_{ACT-MUN}$	139	53.8 ±0.34	55.2 ±0.55	24.0 ±0.67	29.8 ±0.33	1.42 ±0.60	20.5 ±0.47

Lignin is known to be resistant to microbial decomposition and the lignin content of OM is also indicated in empiric equations aiming to predict "substrate degradability" or BVS.

The initial lignin content of WWS_{ACT} treatments (P1, P3 and P4) was similar and around 18% with that of P3 (WWS_{DIG}) lower at 14.2%. The lignin concentration of P1, P3 and P4 increased during composting due to the lower decomposition of lignin relative to the decomposition of cellulose. There was little change in the lignin concentration of P2 over the composting period.

Cellulose is a major constituent of OM and the initial cellulose content of treatments ranged from 40 to 55%. The cellulose content decreased in all compost piles over the composting period and more pronounced over the active phase of composting. Cellulose is known to be highly bio-degradable except when closely associated with lignin in what is loosely termed "ligno-cellulosic" material. Therefore it is not surprising that cellulose decomposition occurred faster than the general rate of OM decomposition, thus being depleted. Surprisingly the final values of cellulose and lignin are close together for all piles except P2, indicating that lignin protects cellulose that are closely associated with it, from decomposition. The hemi-cellulose content of all piles decreased and did so below the detection limit of the analytical method employed in P1 and P2 while dropping below 2% in P3 and P4. The pool of water soluble OM increased in all piles to levels around 20%.

Assuming that ash is conservative (constant mass) and applying the calculations on the values in Table 4.2 results in the absolute decomposition values of OM constituents in Table 4.3 and the graphical presentations in figures 4.10 to 4.13.

In absolute terms, the overall organic matter (VS, LOIOM) decomposition in piles ranged between 27.3% in the pile containing WWS_{DIG} (P2) and the grouping of piles containing WWS_{ACT} of 48.9% (P1), 47.6% (P3) and 49.1% (P4).

Table 4.3 Absolute decomposition (and average daily rate of decomposition) of OM constituents in compost piles over the active (92 days) and curing (38 days) phases of composting

TREATMENT	LIGNIN	CELLULOSE	HEMI-CELLULOSE	SOLUBLE OM
	% of original dry mass average decomposition rate (%.day^{-1})			
P1$_{ACTIVE}$	7.74 0.08	66.5 0.72	67.8 0.74	15.6 0.17
P1$_{CURING}$	10.7 0.28	3.33 0.09	32.2 0.85	(20.7) (0.54)
P1$_{TOTAL}$	18.5 0.14	69.8 0.54	100 0.77	(5.14) (0.04)
P2$_{ACTIVE}$	23.2 0.25	51.7 0.56	100 1.09	32.2 0.35
P2$_{CURING}$	0.42 0.01	(8.09) (0.21)	0.00 0.00	(3.06) (0.08)
P2$_{TOTAL}$	23.6 0.18	43.6 0.34	100 0.77	29.1 0.22
P3$_{ACTIVE}$	26.6 0.29	59.6 0.65	75.5 0.82	34.3 0.37
P3$_{CURING}$	1.05 0.03	9.03 0.24	8.99 0.24	(21.8) (0.57)
P3$_{TOTAL}$	27.6 0.21	68.7 0.53	84.5 0.65	12.6 0.10
P4$_{ACTIVE}$	33.1 0.36	64.7 0.70	84.3 0.92	32.0 0.35
P4$_{CURING}$	(0.53) (0.01)	2.56 0.07	6.60 0.17	(0.77) (0.02)
P4$_{TOTAL}$	32.5 0.25	67.3 0.52	90.9 0.70	31.3 0.24

1: values in parenthesis indicate an increase in absolute concentration

Despite the similar initial lignin content of P1 (18.6%), P3 (18.6%) and P4 (18.1%), lignin degradation differed markedly between these piles. Lignin decomposition in P1 was low and slow throughout but increased from 7.74% over the active phase (at an average rate of 0.08%.day^{-1}) to 10.7% over the curing phase (at an average rate of 0.28%.day^{-1}) to end with a final 18.5% lignin decomposition.

97

Figure 4.10 Organic matter (OM) decomposition in P1$_{ACT-IND}$ over 139 days of composting (assumed: conservation of ash)

Figure 4.11 Organic matter (OM) decomposition in P2$_{\text{DIG-MUN}}$ over 139 days of composting (assumed: conservation of ash)
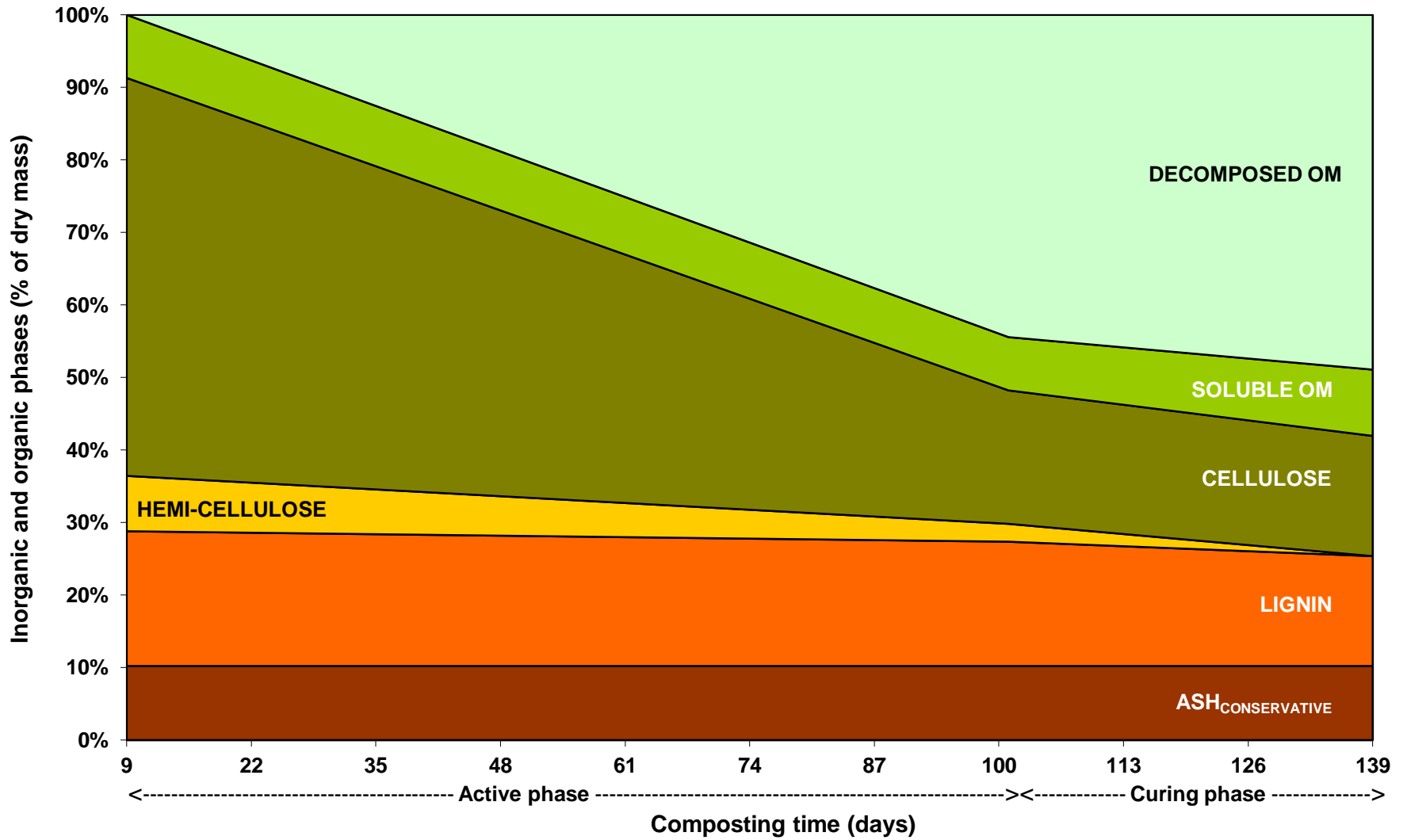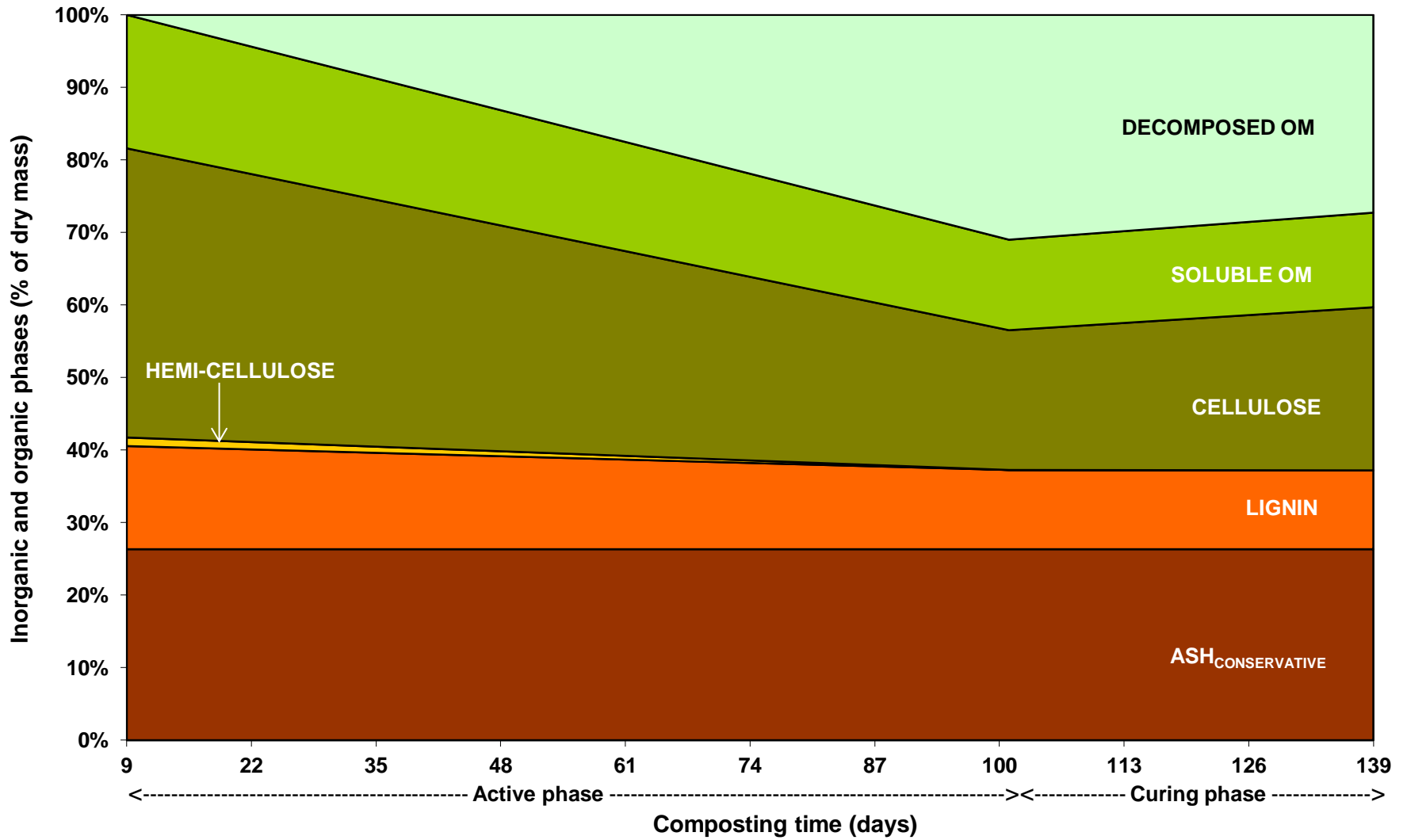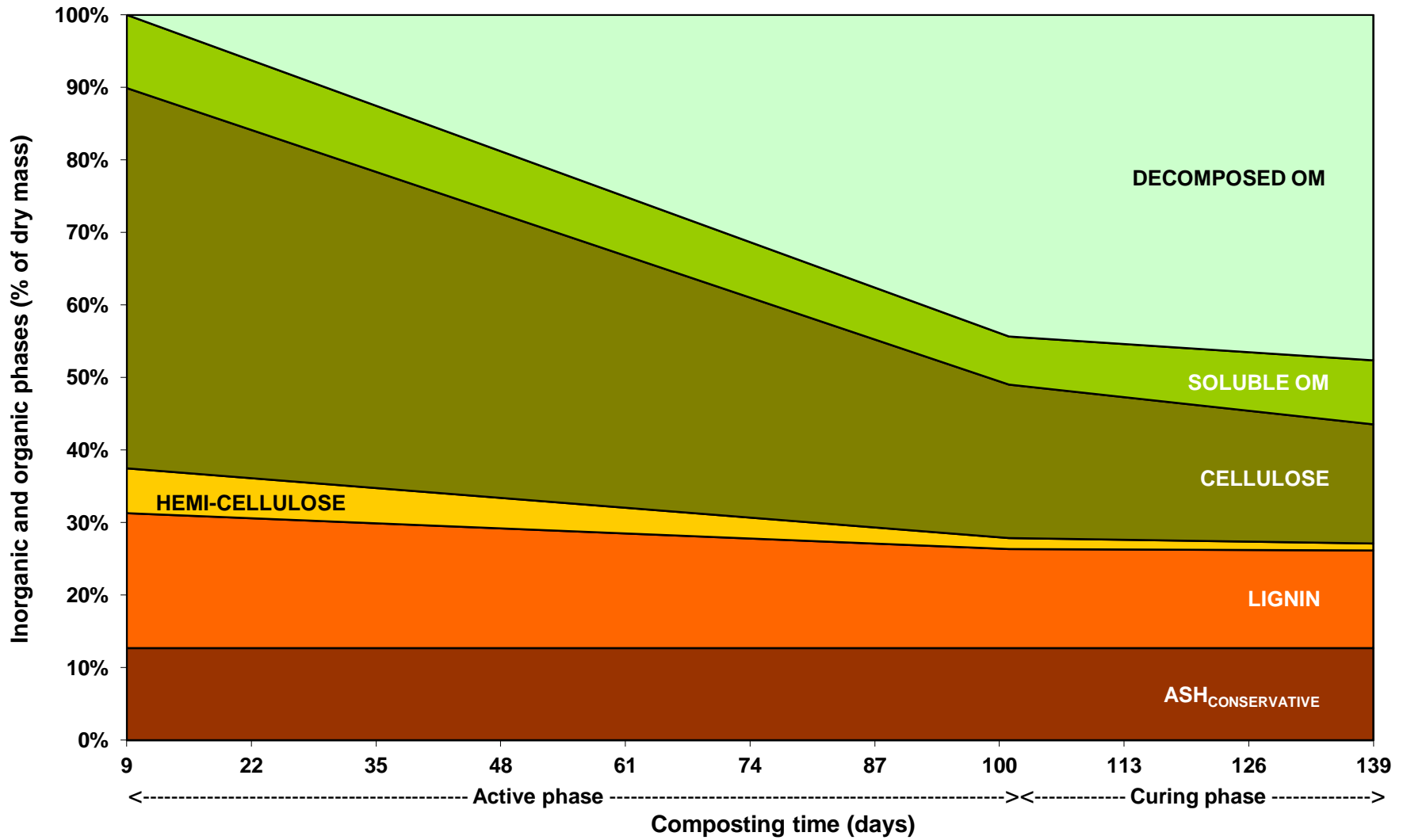
99

Figure 4.12 Organic matter (OM) decomposition in P3$_{ACT-MUN}$ over 139 days of composting (assumed: conservation of ash)
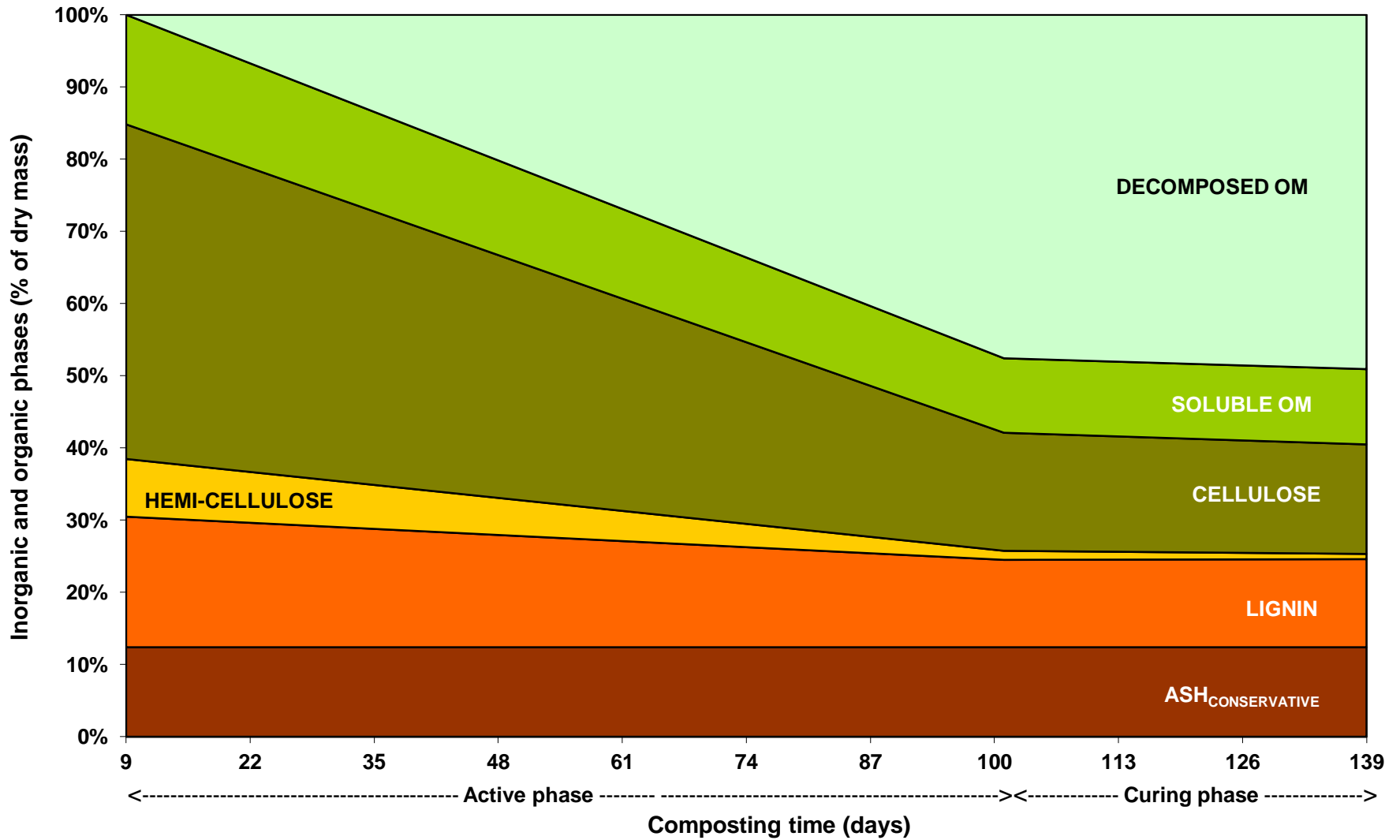
Figure 4.13 Organic matter decomposition in P4$_{\text{ACT-MUN}}$ over 139 days of composting (assumed: conservation of ash)

101

The large increase observed in NaOH extractable C in P1 (Figure 4.7) must have been largely from another source than from lignin decomposition products. Using an average daily rate of decomposition is not meant to suggest that decomposition followed a linear trend but merely to provide a means for comparison and to allow consideration of the different time spans of the composting phases.

The lignin decomposition was much higher and faster over the active phase in P3 (26.6% at an average rate of 0.29%.day^{-1}) and in P4 (33.1% at an average rate of 0.36%.day^{-1}) but slowed down dramatically or came to a complete halt during the curing phase to end with final lignin decomposition values of 27.6% and 32.5% respectively. Lignin decomposition in P2 also largely occurred over the active phase of composting (23.2% at an average rate of 0.25%.day^{-1}).

Cellulose decomposition ranged between 43.6% (P2) and the grouping between 67 to 70% for the piles containing WWS$_{ACT}$ (P1, P3 and P4), again largely over the active phase of composting. The lower lignin and cellulose decomposition observed in P2 can be ascribed to its lower initial lignin and cellulose contents (Table 4.2) and its mesophilic temperature profile (Figure 3.9), actually being in a curing phase from the start of composting.

Hemi-cellulose decomposition ranged between 68 and 100% with P2 in this case exhibiting faster decomposition of its lower initial hemi-cellulose content (1.18%). Again the "unrealistic" ash content values for P2 at the end of the active phase results in a similarly unrealistic increase in organic matter over the curing phase. The soluble OM pool decreased during the active phase of composting and slowly increased during the curing phase in all piles.

4.3.2 Final compost quality characteristics

Some of the final experimental compost quality and stability parameters are shown in Table 4.4. Included are data for commercial compost without sludge in the feedstock

(C1) and commercial sludge based compost (C2) that were analysed for reference purposes.

During the composting process pH played an important role through its influence on microbial populations and nutrient availability. Similarly, the final pH of compost has implications for phytotoxicity (metal mobility) as well as nutrient availability and therefore, the suitability of compost for different applications. pH values between 6 and 8 are optimal for nutrient availability and protection against leaching while metal solubility generally increases with decreasing pH (Thompson *et al*., 2002).

Final pH values were very low for P1 and P2 (< 4.5) and low for P3 and P4 (≈ 5). A sure contributor to this must be the low pH of the sawdust bulking agent used and evidence that the identified need in the beginning of the experiment to add lime to compensate for this low pH was founded. The lime addition accomplished a fair enough pH adjustment to above pH 6.5 for all piles except P2. Although the pH in these piles initially declined over the first part of the active phase, it buffered well during the second part to end the phase between pH 6 and 6.5 (as sampled). Pronounced nitrification during the curing phase produced a large amount of acidity and can be held responsible for the low pH of these final composts. A further addition of lime at the start of curing can be considered for applications where compost pH may be important. The commercial compost tested had fairly optimal pH values around pH 7.

EC is an indicator of the ion concentration i.e. salt content of compost. High salt contents may indicate high plant nutrient content but plants can be damaged through root burn of seedlings, decreased seed germination and at excessively high EC, decreased plant available water and plant nutrient uptake as well as possible phytotoxicity. Very low EC may indicate low fertility levels. Inorganic fertilisers will significantly increase measured EC values (Thompson *et al*., 2002). Compost phytotoxicity has been related to compost $EC > 400$ mS.m[-1] when composts were used as growing substrate (Garcia et al. 1992).

Table 4.4 Final quality and stability parameters of experimental composts compared to commercially available compost that does not contain any WWS (C1[NO WWS]) and commercially available compost that is produced from WWS (C2[WWS]) [± standard deviation – three replicates]

Compost	pH[1]	EC[1] (mS.m^{-1})	Ash[2] (% of dry mass)	C (% of dry mass)	N (% of dry mass)	C:N ratio	CEC (cmol(+).kg^{-1})	WHC (% of total volume)	Porosity (% of total volume)
P1[ACT-IND]	4.52 ±0.02	247 ±4.51	20.0 ±0.24	40.5 ±0.64	2.39 ±0.22	17.0 ±1.77	47.2 ±6.32	46.2 ±0.65	71.7 ±1.49
P2[DIG-MUN]	4.39 ±0.01	478 ±2.08	36.2 ±0.59	27.6 ±0.31	1.91 ±0.09	14.5 ±0.76	27.2 ±3.92	36.9 ±1.37	75.5 ±0.61
P3[ACT-MUN]	5.18 ±0.07	431 ±2.08	24.2 ±0.23	36.8 ±1.42	2.62 ±0.06	14.1 ±0.29	23.9 ±3.00	45.4 ±1.93	76.7 ±2.62
P4[ACT-MUN]	5.15 ±0.04	458 ±1.73	24.3 ±0.69	36.8 ±0.96	2.77 ±0.10	13.3 ±0.75	28.5 ±2.67	41.9 ±0.47	80.5 ±1.88
C1[NO WWS]	6.76 ±0.02	821 ±15.5	51.6 ±1.57	21.4 ±0.70	2.17 ±0.12	9.83 ±0.24	24.7 ±3.42	54.6 ±10.37	64.9 ±6.01
C2[WWS]	7.20 ±0.04	363 ±6.66	35.0 ±4.32	30.2 ±0.54	2.98 ±0.11	10.1 ±0.26	38.2 ±5.26	55.6 ±0.50	65.2 ±4.20

1: as sampled – water content taken into account for 1:10 (mass base) water extract

2: milled samples

The EC of all experimental composts were within an acceptable range although most were on the high side (EC > 400) for certain applications. P1 had the lowest salt concentration (EC of 247 mS.m^{-1}) and lowest ash content (20.0%) coupled with the highest C content (40.5%), highest C:N ratio (17.0) and highest CEC (47.2 cmol(+).kg^{-1}) of all compost tested. C1$_{NO\ WWS}$ had the highest EC (821 mS.m^{-1}) and ash content (51.6%) coupled with the lowest C content (21.4%) and C:N ratio (9.83). This could be an indication of added salts (inorganic fertilisers) and its ash content being above 40% would disqualify this compost for commercial registration in South Africa (Fertilisers, Farm Feeds, Agricultural Remedies and Stock Remedies Act, Act 36 of 1947).

Final experimental composts had good water holding capacities of between 40 and 50% (except P2 = 37%) but lower than the ≈ 55% WHC of commercial composts. Yet, the porosity of experimental composts were between 70 and 80% compared to ≈ 65% for commercial composts, indicating a larger proportion of large pores (and free air space or air filled porosity) for experimental composts. The related and controlling physical parameter would be the particle size distribution or "maximum" particle size as achieved through screening and indicated in South African legislation as a compost standard for certain applications. No screening of the experimental composts was performed and commercial composts were used as packaged and therefore are strictly not comparable in this sense.

Considering the further chemical parameters in Table 4.5, composts generally had comparable N contents between two and three percent with P2 the lowest and the only compost with an N content below 2.00%. P1 had relatively low levels of P (0.30%) and K (0.29%) and possibly due to the industrial i.e. non domestic nature of the ACT-IND sludge.

Table 4.5 Further chemical quality parameters of experimental composts compared to commercially available compost that does not contain any WWS (C1_NO WWS) and commercially available compost that is produced from WWS (C2_WWS) [± standard deviation – three replicates]

Compost	N (%)	P (%)	K (%)	Ca (%)	Mg (%)	Na (%)	S (%)	Fe (%)	Mn (mg.kg⁻¹)	Zn (mg.kg⁻¹)	Cu (mg.kg⁻¹)
P1_ACT-IND	2.39 ±0.22	0.30 ±0.01	0.29 ±0.02	0.61 ±0.02	0.25 ±0.01	0.09 ±0.01	0.31 ±0.01	1.01 ±0.06	266 ±5.60	388 ±27.8	23.3 ±1.82
P2_DIG-MUN	1.91 ±0.09	1.93 ±0.05	0.13 ±0.01	1.72 ±0.12	0.15 ±0.01	0.11 ±0.01	2.38 ±0.02	8.79 ±0.33	1500 ±21.4	2861 ±89.6	517 ±9.51
P3_ACT-MUN	2.62 ±0.06	1.75 ±0.00	0.66 ±0.02	0.90 ±0.03	0.54 ±0.01	0.10 ±0.00	0.38 ±0.02	1.18 ±0.02	375 ±29.4	287 ±14.7	57.6 ±1.04
P4_ACT-MUN	2.77 ±0.10	2.23 ±0.18	0.79 ±0.00	1.31 ±0.08	0.69 ±0.01	0.11 ±0.01	0.50 ±0.00	1.28 ±0.14	505 ±55.3	360 ±24.3	75.3 ±3.30
C1_NO WWS	2.17 ±0.12	1.67 ±0.08	1.92 ±0.19	5.28 ±0.02	0.81 ±0.07	0.36 ±0.03	0.72 ±0.03	2.81 ±0.50	1697 ±138	651 ±35.9	89.2 ±1.09
C2_WWS	2.98 ±0.11	2.43 ±0.04	0.61 ±0.01	3.08 ±0.09	0.59 ±0.02	0.08 ±0.01	0.79 ±0.03	2.05 ±0.06	611 ±9.49	1026 ±31.3	444 ±6.12

The K content of P2 (0.13%) was also very low but clear evidence of the anaerobic digestion can be found in its relatively high S (2.38%) and Fe content (8.79%). The possible commercial use of P2 will be hampered by high levels of Mn (1500 mg.kg^{-1}) and Zn (2861 mg.kg^{-1}) with the maximum content set in the "Guidelines for the utilisation and disposal of wastewater sludge" (Snyman and Herselman, 2006) to achieve **pollutant class a** status is 260 mg.kg^{-1} for Mn, 2800 mg.kg^{-1} for Zn and 1500 mg.kg^{-1} for Cu.

The relatively high levels of especially K (1.92%) and Ca (5.28%) in C1$_{NO\ WWS}$ coupled with its high EC (indicative of salt content) and high ash content point to the possible addition of certain nutrient salts or fertilizers to the organic compost. Due to its relatively "normal" S (the only anion analysed for) content it is unclear what the predominant anion(s) in these salts were but a good guess would be that agricultural lime and KCl was added.

The extremely high Mn content of C1$_{NO\ WWS}$ (1697 mg.kg^{-1}) confirms the addition of dolomitic lime which is known for high levels of Mn, especially in the Pretoria region where large resources thereof occurs. The Mn content of C1$_{NO\ WWS}$ also exceeded the "Guideline" limit for agricultural and certain commercial uses (Snyman and Herselman, 2006). Chemically, the ACT-MUN sludge produced the most balanced compost in both medium (P3) and high levels (P4) of application although both piles also exceeded the maximum Mn limit (as all composts analysed did).

A final measurement of compost stability/maturity was performed by means of a growth chamber bioassay of cucumber seed emergence and seedling development (Table 4.6). The method is designed to detect compost immaturity through oxygen limitations occurring in unstable composts with high levels of CO_2 and low levels of O_2 due to on-going decomposition as well as any phytotoxicity.

Table 4.6 Cucumber seedling emergence and growth for experimental composts compared to commercially available compost produced from WWS (C2$_{WWS}$)

Compost	Emergence (%)	Growth (%)
P1$_{ACT-IND}$	93 (very mature)	93 (mature)
P2$_{DIG-MUN}$	80 (mature > immature)	80 (immature)
P3$_{ACT-MUN}$	93 (very mature)	88 (mature)
P4$_{ACT-MUN}$	34 (immature)	10 (immature)
C1$_{NO\ WWS}$	not tested	not tested
C2$_{WWS}$	61 (immature)	24 (immature)

In the case of P2$_{DIG-MUN}$ the overall "immature" rating for seedling emergence and growth reflects the low pH, medium to high salt content (EC) and likely phytotoxicity of the anaerobically digested sludge compost – indicating a lack in quality rather than stability that needs to be considered in certain applications.

P1 and P3 scored ratings of "mature" to "very mature" and performed the best of composts tested in the bioassay. The higher sludge loading rate in the feedstock of P4$_{ACT-MUN}$ resulted in very low scores for both emergence and growth - a certain indication of immaturity. This can be attributed to insufficient composting/curing time and/or to the higher number of "sludge aggregates" that formed when mixed with the relatively dry sawdust particles (Figure 3.17 and Figure 3.18) that caused localised anaerobic pockets where materials were not fully stabilised due to a lack of oxygen. Seedling emergence and growth were most likely affected by the elevated CO_2 evolution, volatile fatty acid production and oxygen deprivation caused by the further decomposition of the unstable material and possible Mn toxicity (505 mg.kg^{-1}).

The commercial sludge based compost ($C2_{WWS}$) also had very low scores in both emergence and growth of seedlings that could be attributed to Mn (611 mg.kg^{-1}) toxicity and/or general immaturity or instability inducing oxygen deprivation.

4.4   Summary

N transformations during composting have a marked effect on the pH and EC of composting materials and supply information on microbial processes responsible for proton producing (acidifying) and proton consuming (neutralising) reactions that influence pH as well as N mineralisation and immobilisation that influence ion concentration during the phases of composting.

Furthermore, the dissolution/neutralisation reaction of the added lime would also have raised the pH in the initial stages and increased carbonation from elevated CO_2^- levels would have decreased the pH.

As composting proceeded organic acids were further decomposed and CO_2 levels dropped as microbial activity slowed and less degradable organic matter remained. These opposing changes resulted in a buffering of pH over the period from day 45 to day 101 (end of active phase) after the decline from initial feedstock values.

When the temperature fell below 40°C (onset of curing phase), nitrification set in and the conversion of ammonium to nitrate resulted in a more pronounced decrease in pH (H^+ forming) for P1, P3 and P4.

The EC profiles of P1, P3 and P4 remained flat over the first 45 days of composting despite all evidence that prolific mineralisation was occurring. This is a clear indication that most of the ammonium resulting from mineralisation, was assimilated by the growing microbial population and the processes of N mineralisation and N assimilation were in equilibrium.

Only once the easily degradable substrates are depleted towards the end of active composting and mineralisation of the declining thermophilic microbial population occurs, did N mineralisation start to dominate and the EC increased due to an increase in free ammonium. After the onset of the curing phase, the combined processes of re-mineralisation and nitrification resulted in a continued increase in EC up to the end of composting.

The nitrogen in the stabilised digested sludge in P2 was, however, already largely mineralised (+/- 50% of total N) and largely nitrified (+/- 50% of mineral N) and microbial decomposition was far less pronounced in this pile. With its temperature mostly remaining below 40º C, the pile never exhibited a thermophilic phase and the decline in the already low initial pH of P2 was probably caused by early nitrification during the first 45 days of composting. Thereafter, the pH was well buffered and largely remained so to the end of composting indicating that most/all of the ammonium in P2 was already converted to nitrate by the end of day 45.

The relatively high initial EC of P2 declined over the first 45 days of composting, indicating that nitrogen draw down i.e. inorganic N assimilation was dominant over N mineralisation. Thereafter, the EC remained flat from day 45 onwards and then started to increase over the period from day 81 to day 101 due to net N mineralisation from microbial decline.

Ash content is a measure of the mineral component of total solids that can be assumed to be conservative i.e. the total mass of ash in compost piles stays constant over the composting period. Its reciprocal (100 – ash content, %) is a measure of the organic component of total solids referred to as volatile solids (VS). The VS content of composting material is expected to decrease due to microbial decomposition. Assuming that ash is conservative, the absolute decrease in VS content (OM decomposition) could be calculated.

In absolute terms, the overall OM decomposition in piles ranged between 27.3% (P2) and the grouping of piles containing activated WWS of 48.9% (P1), 47.6% (P3) and 49.1% (P4). As an example, the average rate of decomposition in P1 dropped from 0.5 %.day^{-1} during the active phase, to 0.2 %.day^{-1} during the curing phase - demonstrating how the rate of decomposition declines after completion of active composting and during the curing phase.

Chemical transformations reflected the processes of decomposition and mineralisation of OM during composting of activated sludge (P1, P3 and P4). The total C content of these piles showed a pronounced decrease over the active phase and steadier decrease over the curing phase of composting. Contradictory to this, the total C content of P2 increased over the composting period by 3.6%.

The NaOH extractable C fraction of the activated sludge piles (P1, P3 and P4) showed a consistent increase in humic like substances over the active phase as the more resistant compounds like cellulose, hemi-cellulose and lignin were degraded. This was followed by a pronounced decrease in humic-like substances over the curing phase due to an inferred increase in humic acids, the mineralisation of aliphatic "humic like" material and enrichment in aromatic structures typical of the humification process.

The comparatively low content of humic-like substances in P2 decreased over the active phase and remained relatively constant over the curing phase, indicating that humification occurred from the start of composting (as supported by the mesophilic temperature profile).

N consistently accumulated in all compost piles over the composting period and mostly so over the active phase of composting due to the concentrating effect from mass loss during composting. C:N ratios decreased differentially in piles and ended in a fairly narrow band around ratios of 15

111

Biochemical characterisation showed that lignin concentrated in all compost piles, behaving similarly as N content. The cellulose content decreased in all compost piles over the composting period and again more pronounced over the active phase of composting.

The bulk of BVS consists of cellulose and hemi cellulose with a lesser contribution from lignin. It may be that the lignin degradation plays an important role in the process of humification.

Final pH values were low for all experimental composts and compared to commercial compost. Pronounced nitrification during the curing phase produced a large amount of acidity and can be held responsible for the low pH of these final composts.

The EC of all experimental composts were within an acceptable range although most were on the high side (EC > 400 mS.m^{-1}) for certain applications. P1 had the lowest salt concentration (EC of 247 mS.$^{m-1}$) and lowest ash content (20.0%) coupled with the highest C content (40.5%), highest C:N ratio (17.0) and highest CEC (47.2 cmol(+).kg^{-1}) of all compost tested. C1$_{NO\ WWS}$ had the highest EC (821 mS.m^{-1}) and ash content (51.6%) coupled with the lowest C content (21.4%) and C:N ratio (9.83). Final experimental composts had good water holding capacities (WHC) of between 40 and 50% (except P2 = 37%) but lower than the ≈ 55% WHC of commercial composts.

Composts generally had comparable N contents between two and three percent with P2 the lowest and the only compost with an N content below 2.00%. P1 had relatively low levels of P (0.30%) and K (0.29%) and possibly due to the industrial i.e. non domestic nature of the ACT-IND sludge.

The K content of P2 (0.13%) was also very low but clear evidence of the anaerobic digestion can be found in its relatively high S (2.38%) and Fe content (8.79%). The possible commercial use of P2 will be hampered by high levels of Mn (1500 mg.kg^{-1}) and Zn (2861 mg.kg^{-1}).

112

In the bioassay P1 and P3 scored ratings of "mature" to "very mature" and performed the best of composts tested in the bioassay. The higher sludge loading rate in the feedstock of P4$_{ACT-MUN}$ resulted in very low scores for both emergence and growth - a certain indication of immaturity. This can be attributed to insufficient composting/curing time and/or to the higher number of "sludge aggregates" that formed when mixed with the relatively dry sawdust particles that caused localised anaerobic pockets where materials were not fully stabilised due to a lack of oxygen. Seedling emergence and growth were most likely affected by continued CO_2 evolution, volatile fatty acid production and oxygen deprivation caused by the further decomposition of the unstable material and possible Mn toxicity.

In the case of P2$_{DIG-MUN}$ the overall "immature" rating for seedling emergence and growth reflects the low pH, medium to high salt content (EC) and likely phytotoxicity of the anaerobically digested sludge compost – indicating a lack in quality rather than stability.

4.5   References

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Chapter 5: Discussion and conclusions

5.1 Introduction

The study focussed firstly on evaluating the compostability of various types of WWS with eucalyptus sawdust bulking agent. This entailed a composting experiment during which the sludge and bulking agent were selected and characterised, feedstock mixtures formulated according to known principles, certain composting conditions and feedstock characteristics monitored over time, and piles managed through turning, watering, monitoring and sampling.

Secondly, the focus fell on the chemical and biochemical transformations and organic matter decomposition occurring in different piles during composting. These observations also allowed a thorough evaluation of end product (compost) quality. In analysis, these transformations and quality characteristics were as far as possible related to raw material and initial feedstock characteristics, microbial dynamics and environmental conditions during composting.

The aim of this chapter is to briefly discuss the essential components, outcomes and conclusions of the research.

5.2 Discussion

Human generated biological waste and the management thereof is an increasing global problem. Large quantities of domestic and industrial WWS are generated daily by wastewater treatment plants across South Africa. WWS is a predominantly organic material with a high carbon content that contains plant nutrients. Generally composting of organic waste is seen as a very good method of waste stabilisation with considerable environmental advantages.

This led to the hypothesis that WWS can be successfully composted if a suitable bulking agent is added in the correct proportions (feedstock conditioning) coupled with the

116

appropriate process conditions and management. Due to the unique characteristics of different types of WWS, the compostability is expected to differ.

Furthermore, it is hypothesised that during composting of different WWS it can be expected that the chemical and bio chemical evolution will differ and that the end products will be of a differing quality.

In Chapter 1 the principles and conditions for successful composting were elucidated from a variety of scientific sources. Composting refers to the aerobic biological decomposition of organic materials which requires oxygen and produces carbon dioxide, water and heat. Both mesophilic and thermophilic micro-organisms contribute to composting and the process is most rapid when conditions that encourage their growth are established and maintained. Such conditions require a mixture of materials (feedstock) with adequate porosity that allows for enough oxygen through natural ventilation and contains enough water to permit biological activity without hindering aeration. The feedstock also has to provide the required nutrients and energy for microbial activity and growth, including a balanced supply of available carbon and nitrogen as well as a suitable pH range.

In Chapter 2 the quality and safety of compost and the characteristics governing these, were elucidated. Compost can be highly variable in its physical, chemical and microbiological make-up, with definite implications for its quality and functionality in application as well as its safety to plants, animals, humans, and the soil and aquatic environment. These characteristics of compost are intrinsically linked to initial substrate characteristics, management of the composting process and environmental conditions during composting, as well as the maturity of the product. Product standards are necessary to protect public and environmental health and to assure a measure of commercial acceptability.

The heat generated from aerobic composting effectively kills pathogens and weed seeds. It is generally accepted that damp heat is more lethal than dry heat. Compost quality is

117

also closely related to its stability and maturity as it affects a number of its agricultural properties, like phytotoxicity, nutrient availability, suppressiveness against soil-borne pathogens etc. Indicators of compost stability and maturity are important tools to define the end point of composting and to evaluate compost quality.

Chemical properties considered for compost quality and safety assessment includes its content of plant nutrients, heavy metals and hazardous elements as well as cation exchange capacity, electrical conductivity, pH and salinity.

The more complex and resistant organic polymers like lignin and humic substances are considered partially responsible for the physical properties of compost (water holding capacity and cation exchange capacity) and play an important role in the decomposition rates of composts.

Chapter 3 describes the raw material evaluation, feedstock formulation, process management and recorded conditions during a small scale composting experiment of three types of WWS and Eucalyptus sawdust.

Suitable composting mixtures were formulated with reasonable accuracy from a limited number of known raw material characteristics – most importantly water, C and N content. The physical parameters employed to monitor composting (temperature, water content and pile volume), together with the human eye and nose (to observe wetness/dryness and anaerobic conditions), were found to provide adequate for timely management responses like water addition and turning.

Piles of 4 m^3 in size were fairly vulnerable to cold wind and the low ambient temperatures during winter months. Large scale windrows or big static piles should be much more buffered against environmental factors and therefore achieve more sustained high composting temperatures and more "typical" temperature profiles in suitable mixtures. Mechanical windrow turners are expected to largely eliminate the "clumping" observed with manual mixing and turning. The covering of piles with plastic sheeting

118

was necessary for the successful completion of the experiment but did influence the natural draught ventilation and water dynamics of piles.

The relatively high water content of dewatered sludge limits the maximum sludge to sawdust ratio that can be successfully composted. For the activated, dewatered sludge used in this experiment (ACT-IND and ACT-MUN) a maximum sludge-to-sawdust ratio (volume) of about 1:3 would be recommended with dry sawdust containing about 30% water. Treatments containing activated, dewatered sludge (P1, P3 and P4) reached sustained high temperature over the active phase of composting and achieved near 50% volume reduction after 139 days of composting. Judging from the composting conditions resulting from these treatments, activated sludge has a high level of compostability with Eucalyptus sawdust as bulking agent.

The relatively dry DIG-MUN sludge required a considerable amount of initial feedstock water and did not produce a satisfactory composting mixture. P2 reached a maximum temperature of 46°C and a volume reduction of 17%. This low level of compostability can be ascribed to a low pH (4.88) possibly inhibiting microbial activity, a high dry bulk density evidencing low porosity and to the substantially lower C and N content as well as higher ash content of P2 compared to the other composting mixtures. This was a clear indication that the DIG-MUN sludge is already substantially stabilised (and mineralised) and contains insufficient biodegradable organic matter (and therefore insufficient energy) for effective thermophilic composting. Therefore, little benefit can be expected from the composting of DIG-MUN WWS with Eucalyptus sawdust.

Although composting can be monitored more closely and accurately in better controlled experimental conditions like in reactor studies, designing the experiment with a practical composting situation in mind allowed for valuable observations and experiences of the impact that changing and seasonal environmental conditions could have on the process.

In Chapter 4 the temporal changes in certain chemical and biochemical characteristics in the four composting treatments were measured and the final compost quality evaluated. N

transformations during composting specifically have a marked effect on the pH and EC of composting materials and supply information on certain microbial processes that occur during composting.

The pH reflects the nett effect of proton consuming reactions that increase pH and proton forming reactions that decrease pH. These are mostly microbial facilitated reactions like N mineralisation yielding NH_4^+, ammonification yielding NH_3 at high pH or nitrification yielding NO_3^- below 40°C and gaseous denitrification. The pH can also decrease due to the formation of carbonic and organic acids and from the ion exchange of H^+ for NH_4^+ (or other cations) when taken up by microorganisms.

The EC of composting materials reflects a nett increase or decrease in ion concentration and, therefore, dominance of either N mineralisation (ion forming) or N immobilisation (ion consuming), throughout the composting period.

The treatments containing activated sludge (P1, P3 and P4) followed a similar pattern of decreasing pH over the first 45 days of composting (actually a sharp short term decrease followed by an increase to lower than starting levels), a period during which the pH were well buffered from day 45 to the end of the active phase on day 101 and then another decrease in pH from the onset of curing to the end of the composting period. P1, P3 and P4, although further apart, also followed a similar pattern of very little change in EC from the start of composting till the latter part of the active phase when EC values started increasing around day 81 and kept increasing to the end of composting.

These trends read together with the other evidence presented here describe the evolution of a thermophilic population of microorganisms that grew vigorously over the first 45 days of composting while decomposing easily degradable organic matter. The population stabilised over the next 36 days while high rate decomposition of cellulose, hemi-cellulose and to a lesser extent lignin, continued. At the same time most mineralised nutrients (particularly N) were taken up by the growing microbial population. From day 81, the first signs of a population crash were observed in an increase in EC. By day 101

the thermophilic population had disappeared and was replaced with a mesophilic population evidenced in the pronounced mineralisation and nitrification observed during the curing phase of composting.

The nitrogen (and OM) in the DIG-MUN sludge in P2 was, however, already largely mineralised ($\approx$ 50% of total N) and nitrified ($\approx$ 50% of mineral N) and microbial decomposition was far less pronounced in this pile. With its temperature mostly remaining below 40º C, the pile never exhibited a real thermophilic phase and the decline in the already low initial pH of P2 was most probably caused by nitrification (NH_4^+ present and in the mesophile temperature range) during the first 45 days of composting. Thereafter, the pH was well buffered and largely remained so to the end of composting indicating that most of the ammonium in P2 was already converted to nitrate by the end of day 45 and implying that very little freshly mineralised N resulted from composting.

The relatively high initial EC of P2 declined over the first 45 days of composting, indicating that inorganic N assimilation was dominant over N mineralisation (N-negative period). This is often observed in soil after the addition of OM with a too high C:N ratio (e.g. 40) and would not be expected to occur in material with a C:N ratio of 19.3 (P2) which is well below the theoretical optimum of 25 – 30. If you consider that $\approx$ 50% of the total N in the DIG-MUN sludge was already mineralised, the effective "organic" C:N ratio of P2 doubles to $\approx$ 40, thus inducing the N-negative period typical for composting of high C:N material with the advantage that any N-negative period occurs in the compost pile and not in application (Brady and Weil, 1999). The EC of P2 remained flat from day 45 to day 81 when it increased over the next 20 days to day 101 due to nett N mineralisation (and nitrification) following microbial decline. Thereafter, the EC remained flat or slightly decreased, indicating that these processes have come to a stop.

Ash content is a measure of the mineral component of total solids that can be assumed to be conservative i.e. the total mass of ash in compost piles stays constant over the composting period. Its reciprocal (100 – ash content, %) is a measure of the organic component of total solids referred to as volatile solids (VS). The VS content of

121

composting material is expected to decrease due to microbial decomposition. Assuming that ash is conservative, the absolute decrease in VS content (OM decomposition) could be calculated.

In absolute terms, the overall OM (VS) decomposition in piles ranged between 27.3% (P2$_{DIG-MUN}$) and the grouping of piles containing activated WWS of 48.9% (P1$_{ACT-IND}$), 47.6% (P3$_{ACT-MUN}$) and 49.1% (P4$_{ACT-MUN}$) – confirming the observations so far.

Chemical transformations reflected the processes decomposition and mineralisation of organic matter during composting of activated sludge (P1, P3 and P4). The total C content of these piles showed a pronounced decrease over the active phase and steadier decrease over the curing phase of composting. Contradictory to this, the total C content of P2 increased over the composting period by 3.6%.

The NaOH extractable C fraction of the activated sludge piles showed a consistent increase in humic like substances over the active phase as the more resistant compounds like cellulose, hemi-cellulose and lignin were degraded. This was followed by a pronounced decrease of NaOH extractable C over the curing phase due to an inferred increase in humic acids, the mineralisation of aliphatic "humic like" material and enrichment in aromatic structures typical of the humification process.

The comparatively low content of humic-like substances in P2 decreased over the active phase and remained relatively constant over the curing phase, indicating that humification occurred from the start of composting (as supported by the mesophilic temperature profile).

N consistently accumulated in all compost piles over the composting period and mostly so over the active phase of composting due to the concentrating effect from mass loss during composting. C:N ratios decreased differentially in piles and ended in a fairly narrow band around ratios of 15

Biochemical characterisation showed that the initial lignin content of WWS_{ACT} treatments (P1, P3 and P4) was similar and around 18% with that of P3 (WWS_{DIG}) lower at 14.2%. The lignin concentration of P1, P3 and P4 increased during composting due to the lower decomposition of lignin relative to the decomposition of cellulose. There was little change in the lignin concentration of P2 over the composting period. Lignin decomposition varied between 18.5% (P1) and 23.6% (P2) at the lower end and 27.6% (P3) and 32.5% (P4) at the upper end. Lignin decomposition occurred largely over the active phase of composting.

Cellulose decomposition ranged between 43.6% (P2) and the grouping between 67 to 70% for the piles containing WWS_{ACT} (P1, P3 and P4), again largely occurring over the active phase of composting and seemingly limited by the amount of cellulose "protecting" lignin associated with it.

Final pH values were low for all experimental composts. Pronounced nitrification during the curing phase produced a large amount of acidity and can be held responsible for the low pH of these final composts. A further addition of lime at the start of curing or added to the final product can be considered for applications where compost pH may be important.

The EC of all experimental composts were within an acceptable range although most were on the high side (EC > 400 $mS.m^{-1}$) for certain applications. P1 had the lowest salt concentration (EC of 247 $mS.^{m-1}$) and lowest ash content (20.0%) coupled with the highest C content (40.5%), highest C:N ratio (17.0) and highest CEC (47.2 $cmol(+).kg^{-1}$) of all compost tested. P2 had the lowest pH (4.39), highest EC (478 $mS.^{m-1}$), highest ash content (36.2%) and lowest C content (27.6%) of the four experimental composts. Experimental composts had good water holding capacities (WHC) of between 40 and 50% (except P2 = 37%).

Composts generally had comparable N contents between two and three percent with P2 the lowest and the only compost with an N content below 2.00%. P1 had relatively low

123

levels of P (0.30%) and K (0.29%) and possibly due to the industrial i.e. non domestic nature of the ACT-IND sludge. The K content of P2 (0.13%) was also very low but clear evidence of the anaerobic digestion could be found in its relatively high S (2.38%) and Fe content (8.79%). The possible commercial use of P2 will be hampered by high levels of Mn (1500 mg.kg^{-1}) and Zn (2861 mg.kg^{-1}).

In the bioassay P1 and P3 scored ratings of "mature" to "very mature" and performed the best of composts tested. The higher sludge loading rate in the feedstock of P4 resulted in very low scores for both emergence and growth - a certain indication of immaturity. Seedling emergence and growth were most likely affected by continued CO_2 evolution, volatile fatty acid production and oxygen deprivation caused by the further decomposition of the unstable material and possible Mn toxicity.

In the case of P2$_{DIG-MUN}$ the overall "immature" rating for seedling emergence and growth reflects the low pH, high salt content and likely phytotoxicity of the anaerobically digested sludge compost – indicating a lack in quality rather than stability.

5.3   Conclusions

The main conclusions derived from this study are that:

- Dewatered, activated sludge has a high level of thermophilic compostability with Eucalyptus sawdust as bulking agent. Mixture formulation is primarily limited by water content and no initial water addition is required, making it more water efficient than composting dried sludge. The C:N ratio and pH of mixtures needs to be considered to be within the acceptable range. Activated sludge contained sufficient bio-chemical energy (i.e. BVS) for successful thermophilic composting. Large scale windrow composting combined with mechanical turning (typical of large scale composting) should take care of the negative impact on composting temperature from adverse environmental conditions and the aggregate formation experienced in the small scale experiment.

- Dried, digested sludge has a low level of compostability with Eucalyptus sawdust as bulking agent. Mixture formulation requires initial water addition that can be limited

124

by the WHC of the composting mixture. Although the C:N ratio of the mixture was low, the digested sludge did not contain sufficient bio-chemical energy (i.e. BVS) for successful thermophilic composting. The high EC and ash content and the low total C and N content of the digested sludge gave indications of a high level of stability/mineralisation and therefore, low level of bio-chemical energy. This can be verified through determining the organic C and the N speciation (including organic N) in the sludge when predicting compostability.

- The chemical and bio-chemical evolution in treatments largely supported the physical observations and gave very good insight into the microbial processes that drives the different phases of composting. In the final analysis, differences in the observations between treatments could be linked to the quality and/or stability of the final compost. The final compost quality was also closely linked to the quality of the raw materials (especially the WWS) used.

- Overall, dewatered ACT-MUN sludge and eucalyptus sawdust produced the compost of highest quality and stability/maturity in a volume ratio of 1:4 (sludge:sawdust) and an unstable/immature compost in a volume ratio of 1:3 (sludge:sawdust).

- Dewatered ACT-IND sludge and eucalyptus sawdust produced an interesting compost of high stability/maturity with the highest content of humic substances and highest CEC, but with a low content of certain plant nutrients and low pH. Increasing the sludge:sawdust ratio from 1:6 closer to 1:4 (volume) will enhance the chemical quality of this good compost.

- Dried DIG-MUN sludge and eucalyptus sawdust produced a stable compost of very low pH, relatively low fertility, poor physical character and high risk of phytotoxicity (from the low pH and high levels of certain elements in the sludge). The only real value composting imparted to the already stable digested sludge was a further curing of the material which entailed limited mesophilic decomposition, a slight concentration of nutrients and other elements in the material and the advantage typical for composting of high C:N material that any N-negative period will occur in the compost pile and not in soil application.

125

- All the above instances of low pH and the resultant risk of phytotoxicity can be alleviated by the addition of agricultural lime during the composting process or to the end product.

Composting is a great option for WWS treatment that deserves a lot more attention in South Africa – especially for WWS with a good chemical quality. Composting is a fairly adaptable technology with a lot of composting system choices and good process control is possible from a limited number of important material and process characteristics and parameters.

5.4 References

BRADY, N.C. and WEIL, R.R., 1999. The nature and properties of soils, 12th edition, Prentice Hall, New Jersey.

Appendix A: Further details pertaining to Materials and Methods

1. Composting mixture formulation (Chapter 3)

The general equation for determining the water content of a composting mixture from the water content of the constituents is:

$$G = \frac{(Q_1 \times M_1) + (Q_2 \times M_2) + (Q_3 \times M_3) + \cdots}{Q_1 + Q_2 + Q_3 + \cdots}$$

in which:

Q_n = mass of material n ("as is", or "wet mass");

G = moisture goal (%); and

M_n = water content (%) of material n.

For two materials, the general equation can be simplified and solved for the mass of a second material (Q_2) required in order to balance a given mass of the first material (Q_1) for a specific mixture water content:

$$G = \frac{(Q_1 \times G) + (Q_1 \times M_1)}{M_2 - G}$$

The general equation for determining the C:N ratio of a composting mixture from the C and N content of the constituents is:

$$R = \frac{Q_1(C_1 \times (100 - M_1) + Q_2(C_2 \times (100 - M_2) + Q_3(C_3 \times (100 - M_3) + \cdots}{Q_1(N_1 \times (100 - M_1) + Q_2(N_2 \times (100 - M_2) + Q_3(N_3 \times (100 - M_3) + \cdots}$$

in which:

R = C:N ratio of compost mixture;

Q_n = mass of material n ("as is", or "wet mass");

C_n = carbon (%) of material n;

N_n = nitrogen (%) of material n; and

M_n = water content (%) of material n.

This equation can also be solved for a mixture of two materials, knowing their carbon, nitrogen, and water contents, the C:N ratio goal, and specifying the mass of one ingredient. By simplifying and rearranging the general equation, the mass of the second material required would be:

$$Q_2 = \frac{Q_1 \times N_1 \times \left(R - \frac{C_1}{N_1}\right) \times (100 - M_1)}{N_2 \times \left(\frac{C_2}{N_2} - R\right) \times (100 - M_2)}$$

2. Determination of C and N content (Chapters 3 and 4)

Laboratory analyses of material samples (3 replicates) consisted of the following:

- Total C and N content were determined with a Carlo Erba NA 1500 C/N/S Analyzer:
    - The sample is used directly (in powder form) using approximately 10 to 15mg sample weighed into a tin foil container for each determination. The method is a dry oxidation method generally known as the Dumas method.
    - The sample and tin container are ignited at high temperature (1020ºC) in oxygen (on a chrome oxide catalyst) to produce carbon dioxide, nitrogen gas and oxides of nitrogen (plus other oxides etc.). The gases produced pass through silvered cobalt oxide (to remove oxides of S and halogens etc.) and a column of copper (550ºC), which reduces the oxides of nitrogen to nitrogen gas (and removes excess free O_2).
    - After removal of water vapour by a trap of anhydrous magnesium perchlorate, the N_2 gas and CO_2 are finally separated by gas chromatography using a helium carrier gas and detected by a thermal conductivity detector.
    - The instrument is calibrated against a pure organic compound of known composition. The concentrations of C and N obtained are the concentrations in the sample as analysed, but these values may be converted to a dry basis if necessary. Note that the values obtained after conversion to a dry basis will often be higher than values obtained from direct determination on oven-dried samples, due to probable losses of N in the form of ammonia and C in the form of CO_2 during oven drying (Institute of Soil, Climate and Water laboratories).

3. Phytotoxicity bioassay (Chapter 4)

Seedling emergence and relative growth (vigour) in potting media was determined through direct seeding using distilled water as the moisture source.

- Cucumber seeds were planted in a blend of 50% compost and 50% No. 2 grade vermiculite. Two controls, pure vermiculite (negative control) and a soilless commercial potting media (positive control) were employed.
- The emergence % was calculated from the ratio of emerged seedlings to the number of seeds planted. Relative seedling health and vigour was evaluated through considering seedling turgor, hypocotyl length (stunting) and relative distortion or shape of cotyledons.
- From these indicators the maturities of the composts were rated according to the limits set in Table 4.1.
- Cucumber is considered a good indicator species for this test because of its high salt tolerance, large seed size, distinct cotyledon shape, and intolerance to volatile fatty acids (Thompson *et al.*, 2002).

Table A.1 Maturity Indicator Rating (Thompson *et al.*, 2002)

Test Parameters	Very Mature	Mature	Immature
Emergence (%)	> 90	90 – 80	< 80
Seedling Vigour (%)	> 95	85 – 95	< 85

4. Cation exchange capacity (Chapter 4)

Laboratory analyses of samples (3 replicates) consisted of the following:

- Cation Exchange Capacity (CEC) was determined through the displacement of cations on exchange sites in the sample with NH_4^+ cations by leaching with an ammonium acetate buffer solution (1mol.dm^{-3}, pH 7).
  - The sample is then washed with ethanol to remove all traces of the NH_4^+ not held by exchange sites.

130

- Following this step, the sample is leached quantitatively with a replacement solution of potassium chloride. The replacement solution is analysed with distillation and titration techniques for ammonia. CEC is expressed as cmol(+).kg^{-1} dry mass (Institute of Soil, Climate and Water laboratories).

5. ICP determination for elemental analysis (Chapter 4)

Laboratory analyses of samples (3 replicates) consisted of the following:

- An aliquot of the perchloric/nitric acid digest solution (page 79) is used for the ICP-OES (Inductively Coupled Plasma Optical Emission Spectrometric, Varian Liberty 200 instrument) determination of the Ca, Mg, P, K, Na, Fe, Zn, Mn and Cu content of samples. Each element is measured at an appropriate emission wavelength, chosen for high sensitivity and lack of spectral interferences. The wavelengths used were: Mg (383.826nm), Ca (422.673 and 317.933nm), P (213.618nm), K (769.896nm), Na (589.592nm), Fe (259.940nm), Mn (257.61nm), Zn (213.856nm) and Cu (324.754nm). The instrument is calibrated against a series of standard solutions, containing all the elements of interest in the proportions found in typical compost samples (Institute of Soil, Climate and Water laboratories).

131