ABSTRACT

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Composting is a reasonable option for managing swine wastes prior to land application. However, the process is protracted, and the loss of nutrients can be considerable, diminishing the usefulness as a plant nutrient source. For these reasons, reducing composting time has been of interest. The objectives of this research were to compost swine waste feedstocks for various lengths of time, use them as soil amendments in a field study, and subsequently relate the compost characteristics to plant response and changes in soil characteristics. Additionally, a laboratory nitrogen mineralization study was conducted to determine mineralization rates of the amendments.

Swine manure solids and Paulownia branches/leaves were co-composted for 0, 3, 6 and 15 weeks. For the field study, the composts and manure were used as soil amendments; fertilized and unfertilized soils were the controls. The uncomposted feedstock-amended soil produced the greatest sorghum stover yield, although grain yields from all treatments were statistically equivalent. With respect to soil physical properties, the effect of treatment on soil bulk density was statistically significant, but for soil impedance and aggregate stability, treatment was not statistically significant. The water holding capacity was not statistically affected by treatment at either field capacity or wilting point. Compared the controls, the reduction in plant available water induced by the manure-amended soil was statistically significant. Due to the minimal effects of soil treatments on soil and plant response, relating amendment characteristics to field response was not feasible.

The results from a nitrogen mineralization study did not confirm the projected mineralization rates for the amendments, which were hypothesized from the literature. As the amendment application rates were based upon the projected mineralization rates, the field loading rates may have been low. However, the nitrogen mineralization dynamics in the laboratory and in the field may have been dissimilar.

Co-Composting and Characterization of Swine Waste Solids and Its Use as a Soil Amendment in a Field Study: The Effects of Duration of Composting

by

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DEDICATION

I dedicate this dissertation
to my brother, Noah,
whose perseverance in life
dwarfs all my efforts
in completing
my PhD.

BIOGRAPHY

Life dances and you have to dance with it, whether it is taking you on a wonderful ride or is stepping on your toes . . . . Life will move you in the rhythm and direction of its own . . . .

Phillip Moffitt

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Chapter 1
Introduction

1.1 Overview

Composting is defined as the controlled aerobic decomposition of organic materials. The primary objective of the process is to produce a homogeneous, "stable", humified product, stability connoting that it can be used as a soil amendment without being deleterious to seed germination and plant growth. Being free of pathogens (plant and animal) and weed seeds, and ease of handling are other desirable qualities.

Stable composts made from agricultural, industrial or municipal solid wastes have been shown to enhance soil properties and improve crop yields, while unstable products can have deleterious effects on plant growth. Producing a stable product is a relatively protracted process, especially using the "windrow" method, which is a technique whereby the compost feedstocks are laid out in rows, aerobiosis within the piles is maintained passively, and temperatures cannot be precisely controlled. Nutrient losses, via volatilization and leachate runoff, occur during composting, generally increasing with composting time. In an attempt to minimize these losses and economic costs (by reducing composting time), unstable/less humified composts have been studied for use as soil amendments. At the time that the present study was initiated, data from laboratory/greenhouse studies existed indicating that fully stable compost may not be necessary in all agricultural applications. However, there was a paucity of data concerning the effects of compost at different stages of decomposition on plant response in field trials.

While characterizations of composts by chemical and physical analyses have been the convention, thermodynamic characterization has not. Preliminary work, by the present author, indicated that calorific values of compost appeared to be a characteristic indicative of duration of decomposition, and consequently might be useful in relating soil properties and plant response to compost quality. The present research was conducted to better understand the relationship between compost quality, soil characteristics and plant response in a field trial. This work was an expansion of extant research involving the use of swine wastes as container growth media.

1.2 Background

1.2.1 Swine Wastes

North Carolina is the second largest swine producer in the US, with a population of approximately 9.5 million head in June, 2007 (Agricultural Statistics Division, 2007). With each hog excreting up to approximately 2 tons of manure per year (Lander et al., 1998), a variety of strategies has been used to address the waste management issues. Composting the wastes is considered to be a viable option (Williams, 2006).

Land application is a common method of manure utilization because of the value of the nutrients and organic matter. However, manures contain pathogens that pose risks for contamination of drinking water supplies and shellfish beds; human health is also at risk if manures are applied to crops inappropriately (USDA, 1999). In North Carolina, management of farm animal wastes is regulated by State regulations 15A NCAC 02T.1300, which include land application guidelines (such as for nutrient management). Federal law, 40 CFR (Code of Federal Regulations) Part 503, regulates waste management strategies for human wastes, with the focus on minimizing the risk of disease transmission; composting is designated as one option for processing the wastes. There are no requirements for reducing pathogenic microorganisms or disease vectors in animal manures prior to land application.

In the present investigation, windrow composting was used as the waste management approach for processing swine wastes, specifically manure solids. In hopeful anticipation that disease risks from animal wastes would be regulated to a similar degree as human wastes, one objective in this procedure was to follow the federal regulations (40 CFR Part 503) to produce a composted product that would have minimal risk of disease transmission when land applied.

1.2.2 Bulking Agents

Swine waste management systems employed in North Carolina are usually water-wash systems that result in wastewater with low suspended solids concentration. When composting manures, a secondary organic material, termed bulking agent, is often added to improve the efficiency of the process. Bulking agents are used to absorb water, provide structural support to maintain aerobic conditions, and to adjust carbon and nitrogen concentrations.

In the present investigation, peanut shells (wastes from peanut processing) were

originally selected as the bulking agent to continue using the same feedstocks as in previous research. However, availability of the material became an issue when peanut shells were unable to be procured. Leaves and branches from Paulownia trees became an option for a bulking agent, and were subsequently selected. These trees grow rapidly and have the ability to regenerate from their own root. Because of these qualities, plus their ability to take up relatively large quantities of nitrogen, copper and zinc from the soil, the trees have a potential for use as a swine waste utilization species (Bergmann et al., 1997).

Chapter 2
Literature Review

2.1 Organic Matter

Organic matter is an important component for soils, and is the primary resource for nutrients for soil microorganisms and plants. Exogenous sources of organic matter are added to soils to improve soil characteristics, to provide phytonutrients, and consequently to improve plant growth (Somani et al., 1975; Barzegar et al., 2002). The enhanced soil properties and plant growth are due to the inherent properties of the OM (Hempfling et al., 1990; Varianini et al., 2001), such as acting as a pH buffer due to a high ion exchange capacity and facilitating nitrate uptake in plants (Piccolo et al., 1992; Cacco et al. 2000), and also because of the increased microbial activity due to the availability of nutrients. The stimulated microbial proliferation (Nishio et al., 1983) results in elevated production of exudates, which affect the soil structure (aggregation) and therefore soil physical properties (Tisdall, 1996), which are important factors in seed germination and plant growth (Pagliai et al., 1993; Schneider et al., 1985; Barker, 2001). Increased microbial activity is associated with increased root growth and plant uptake of nutrients (Schnürer et al., 1985; Perfect et al., 1990a; Caravaca et al., 2002). The magnitude of the effects of organic matter additions are dependent upon the chemical and physical characteristics of the organic matter, the soil characteristics and plant species (Abd-el Naim et al., 1975; Barker, 2001; Chambers et al., 2002), as well as the length of time that the amendment is in the soil (Barker, 2001).

2.2 The Composting Process

2.2.1 General

When used as a soil amendment, fresh organic matter, whether its origin is agricultural, industrial or municipal, can have deleterious effects on plant growth (Wong et al., 1985b; Baca et al., 1990). Readily available pools of nutrients in the exogenous OM will stimulate soil microbial activity potentially creating oxygen deficiencies for plants; if the OM does not provide sufficient nitrogen, the available nitrogen will be primarily incorporated into microbial biomass and not be

available to plants. Extant chemical moieties in the OM and intermediate products of decomposition may be phytotoxic, inhibiting seed germination and plant growth. The presence of weed seeds and plant pathogens are undesirable and the existence of disease vectors may pose human health risks.

Composting, which is a controlled aerobic decomposition process, is a technique to ameliorate the aforementioned problems. In a properly composted mixture, a heterogeneous raw organic mixture is transformed into a more compositionally uniform product. The phytotoxic effects are often reduced via microbial catabolism, and the high temperatures achieved during the process minimize the problems associated with weed seeds and plant pathogens and potential health hazards of animal pathogens. With high water content feedstocks, such as animal manures, the composted product is drier than uncomposted OM and consequently facilitates further handling and distribution.

Composting is a microbially mediated process and therefore optimizing the process means optimizing the microbial environment for efficient decomposition. Nutrients, oxygen and water are the essential components. Traditionally, the nutrients have focused on carbon (C) and nitrogen (N) as these macronutrients are often out of balance for optimal decomposition and can readily be manipulated. Phosphorus (P) is also a macronutrient, and controlling C:P ratios has been found to improve decomposition rates in phosphorus deficient feedstocks, such as municipal solid wastes (Brown et al., 1998).

Oxygen is necessary for microbial aerobic respiration, which is more efficient (ie, higher microbial yield) than anaerobic processes, and also reduces the evolution of obnoxious gases (Zhu et al., 1999). Maintaining aerobic conditions means that the composting mass must be sufficiently porous to allow airflow throughout the duration of composting. Moisture content is inextricably related to oxygen levels, since sufficient moisture is necessary for microbial activity (minimum of approximately 15%), yet too much water will create anaerobic conditions. A moisture range of 40-60% has been deemed optimum (Pal et al., 1975; Cabrera et al., 1994).

2.2.2 Bulking Agents

Bulking agents are used to modify the chemical and physical characteristics of the composting mass. In the case of high moisture swine manure solids, rigid materials will provide

structural support for the composting piles, and allow aerobic conditions to prevail for extended periods during the composting process. Bulking agents can be used to regulate decomposition dynamics by adjusting the carbon and nitrogen concentrations, since the carbon-to-nitrogen ratio of the mixture influences the efficacy of the composting process. They can also adjust the moisture content by absorbing water to initially create aerobic conditions within the composting mass.

Non-technical factors are just as important since the final product of composting is not considered a high value product. Other issues include material cost and availability and associated transportation costs. There is not a "best" bulking agent for composting a specific waste. All of the aforementioned variables have to be considered when selecting them.

2.2.3 The Process

2.2.3.1 The Basics

Once the proper initial composting conditions have been created, microbial activity is stimulated, which causes a rise in temperature of the composting mass. As the temperature increases from ambient into the thermophilic range (> 45^{O} C), a succession of different microbial communities predominates (Godden et al., 1983; Riffaldi et al., 1986; Horwath et al., 1996b). Microbial activity diminishes at high temperatures, due to the heat inactivation of enzymes (Ladd et al., 1972) and the heat intolerance of thermophilic microbes, which begin at temperatures exceeding 60^{O} C (Chang et al., 1967), with optimum composting temperatures being approximately 55-60^{O} C (MacGregor et al., 1981; Sikora et al., 1985; Strom, 1985). The compost temperature remains at a high level until the organic substrate becomes sufficiently depleted that insufficient nutrients are available to sustain the high microbial activity. Consequently, the microbial activity declines, the decomposition rate decreases, and the temperature decreases into the mesophilic range (15-45^{O} C). The decomposition of OM primarily occurs in the thermophilic temperature range (Horwath et al., 1995; Sanchez-Monedero et al., 1999; Bidegain et al., 2000; García-Gómez et al., 2003 & 2005) and the high temperatures achieved within the composting mass inactivate pathogenic microbes and weed seeds (Burge et al., 1981; Churchill et al., 1995; Meekings et al., 1996). As the composting process continues, a heterogeneous mass of organic matter becomes further transformed into a more compositionally uniform product (Hsu et al., 1999).

In OM there are often several pools of nutrients, defined by their bioavailability, from readily available to recalcitrant (Knapp et al., 1983; Reinertsen et al., 1984; Adani et al., 1997; Bernal et al., 1998a & c; Benito et al., 2005). As decomposition progresses, the readily biodegradable nutrients are consumed first, leaving the more recalcitrant forms, as well as labile forms being converted into more refractory ones. Consequently, as the composting process proceeds, a slower release nutrient system evolves (Kirchmann, 1990; Keeling et al., 1994b; Gale et al., 2006).

2.2.3.2 Pathogens

Pathogenic microorganisms are ubiquitous in human and animal wastes (Strauch, 1991). Therefore, minimizing the risk of disease transmission prior to land application of these wastes is a primary goal of composting.

The elevated temperatures that evolve in compost piles are the primary mechanism for pathogen destruction during the composting process; microbial competition and antagonism, and production of antibiotics and ammonia during the decomposition process have also been attributed to reductions in viable pathogens (Wiley, 1962; Ward et al., 1977; Burge et al., 1987). Although high temperatures are necessary for pathogen destruction, the duration at these temperatures is critical (Wiley et al., 1962; Burge et al., 1981).

Pile turning is integral to the windrow composting process. Pile turning promotes uniform decomposition, redistributes pathogens to ensure exposure to high temperatures, breaks up clumps of OM that would hinder pathogen destruction and enhances aerobic conditions within the pile by re-establishing the material's structure and restoring air flow channels that may diminish during OM decomposition (Haug, 1980; Barrington et al., 2002).

The US EPA established regulations (40 CFR 503) for reducing the risk of disease transmission of wastewater residuals prior to land application; composting was considered to be a viable alternative. To minimize the risk of disease transmission both pathogen and vector attraction reductions are necessary. For maximum pathogen reduction, EPA established processing criteria (Process(es) to Further Reduce Pathogens - PFRP), which are processes equivalent in their ability to reduce pathogen concentrations. For windrow composting, the technique used in the present study, to be an equivalent PFRP, compost pile temperatures must

remain at least 55^{O} C for 15 days and within that time period the pile must be turned at least 5 times. Additionally, vector attraction must be reduced before or simultaneously with pathogen reduction. One option is to maintain compost pile temperatures above 40^{O} C for at least 14 days, which would be accomplished simultaneously with pathogen reduction (EPA, 1992).

For composting, the temperature/time criteria are based on the most thermally resistant microorganisms (eg, bacteriophage f2, helminth ova) (Burge et al., 1981). Destruction of the most resistant pathogens ensures destruction of the less heat resistant microbes. Five pile turnings ensure appropriate redistribution of pathogens throughout the compost piles and exposure of the pathogens for sufficient time at high temperatures to reduce pathogen densities (*Salmonella* sp., enteric viruses and helminth ova) to below detectable limits (Haug, 1980; EPA, 1992). The criteria for pathogen and vector attraction reductions, previously described, were the procedures followed for composting in the present study.

2.2.3.3 Risk Assessment

Disease transmission from pathogen-contaminated wastes (PCW) can occur directly or indirectly. Risk assessment, for federal regulations 40 CFR 503, was based on different exposure pathways. Direct exposure pathways include inadvertent contact, handling soil or plants where the PCW has been applied or inhalation of airborne microbes via product aerosols, dust, etc. Indirect exposures include consumption of pathogen contaminated crops or other foodstuffs that have been contaminated by contact with product, consumption of milk or other animal food products from animals grazing in pastures or being fed crops grown on product amended soil, ingestion of drinking or recreational waters that have been contaminated by runoff from product amended fields or by migration of pathogenic microbes into groundwater, consumption of inadequately cooked pathogen-contaminated fish from contaminated waters, and contact with product or pathogens due to transport via vectors (eg, rodents, insects) (EPA, 1992).

Two categories of pathogen reduction procedures were developed based on the need for particular levels of pathogen reduction. The objective of Class A pathogen (PFRP) reduction is to reduce pathogens (enteric viruses, pathogenic bacteria and viable helminth ova) to below detectable levels (EPA, 1992). Class B pathogen (PSRP) reduction allows higher concentrations that are unlikely to pose a threat to public health and the environment under specific use

conditions (EPA, 1992). Additionally, certain vector attraction methods are required along with pathogen reduction. Site restrictions are imposed to minimize the potential for human and animal contact with Class B product for a period of time following land application until environmental factors have further reduced pathogen levels. There are no site restrictions for land applying Class A product (EPA, 1992).

The public and animals can be protected by 1) pathogen reduction, 2) vector attraction reduction and 3) site restrictions. EPA uses a combination of these methods to minimize the risk of disease transmission to maximum extent.

2.2.4 Chemical Changes During the Composting Process

2.2.4.1 Carbon and Nitrogen Transformations

Organic matter serves as a source for biomass synthesis, biomass byproducts and redox reactions. Microbes use organic carbon as an energy source, and for biomass synthesis. The catabolic metabolism of organic carbon generates carbon dioxide, water and energy.

Organic nitrogen is used as electron donor molecules (oxidizing agents) and is converted into ammonium (ammonification), an inorganic form of nitrogen. Subsequently, the ammonium is assimilated by microbes, being re-transformed into the organic form, or further oxidized into nitrate (nitrification). The transformations of organic carbon and nitrogen to inorganic forms (mineralization) are a normal consequence of aerobic respiration.

Microbes utilize carbon at a greater rate than nitrogen to replenish those nutrients in the same ratio that exist in the cellular biomass. Bacteria have C:N ratios of 3.7-17, with most investigators reporting ratios between 3.7 and 6.7; for fungi, a range of 4.5-44 has been reported (Paul et al., 1996; Bloem et al., 1997). While microbial efficiency of converting organic carbon and nitrogen to microbial biomass is dependent upon substrate, environmental conditions and specific microorganism, empirical observations have indicated that a C:N range of approximately 20-30 optimizes microbial metabolism (Golueke, 1992; Huang et al., 2001). However, Eiland et al. (2001a) reported greater decomposition rates of cellulose and hemicellulose with compost having an initial C:N ratio of 11, compared to those with C:N ratios of 35-54.

Above the optimum C:N threshold (and assuming nutrient availability and oxygen are not limiting), a deficit of nitrogen exists. Consequently, nitrogen is predominantly assimilated. As

decomposition continues, the C:N ratio decreases, and microbes continue to utilize carbon and nitrogen in the approximate ratios of 20-30. When the C:N ratio is reduced to less than the optimum, the excess organic nitrogen is converted to ammonium (and then to nitrate). An inverse relationship exists between mineralized nitrogen and C:N ratio (Rubins, 1942; Enwezor, 1976; Hartz et al., 2000). The critical C:N ratio above, which nitrogen assimilation dominates and mineralization is minimal, has been reported to be between 15 and 40 (Rubins, 1942; Enwezor, 1976; Stockdale et al., 1995; Davet, 2004).

However, the dynamics of carbon and nitrogen transformations are highly dependent upon the characteristics of the organic matter. For example, Rowell et al. (2001) amended soil with 4 different biosolids, and all caused net nitrogen immobilization for 14 days, with subsequent net mineralization (the sampling points were initial, 14 days, 145 and 397 days). The C:N ratios ranged from about 8-13. However, wheat straw with a C:N ratio of 50 followed the same trend, although needle litter, with a C:N ratio of 52, caused net immobilization the entire incubation period; paper fines with a C:N ratio of about 178, also caused net immobilization the whole incubation period.

Organic matter is decomposed at different rates due to varying degrees of bioavailability of nutrients. The varying rates are indicative of disparate pools of carbon and nitrogen (Ajwa et al., 1994; Tachimoto, 1995; Bernal et al., 1998c). Decomposition rates decrease as the more labile fractions are metabolized first, followed by the more recalcitrant fractions. Rates of decomposition are often quantified by measuring the production of inorganic carbon (carbon dioxide) and nitrogen (total ammoniacal nitrogen and nitrate). Rates of carbon and nitrogen mineralization decrease the longer the composting process proceeds (Robertson et al., 1995; Bernal et al., 1998c; Benito et al., 2005).

2.2.4.2 Humic Substances

One objective of composting is to produce a more homogeneous humified product. Humification is the conversion of more labile forms of organic matter into more recalcitrant forms, called humic substances, which are more complex, higher molecular weight, polymers with significant aromatic character. These transformations occur through a complex of polymerization/polycondensation reactions. Humic substances are colloidal molecules,

comprised of humic and fulvic acids and humin, each one operationally defined by its solubility. Humic acids are soluble in aqueous alkaline solutions but insoluble in aqueous acidic solutions, fulvic acids are soluble in both aqueous alkaline and acidic solutions and humin is insoluble in both.

While humification is integral to OM decomposition dynamics, humification during composting has been inconsistently observed. Humic substance content has been reported to increase (Inbar et al., 1990; Miikki et al., 1994; Ouatmane et al., 2000), remain invariant (de Nobili et al., 1988; Chefetz et al., 1996) and decrease (Garcia et al., 1991b; Miikki et al., 1994; Adani et al., 1999), the changes apparently being dependent upon the feedstock (Miikki et al., 1994; Chefetz et al., 1996; Bernal et al., 1998b). However, a primary reason for inconsistency is the fact that humic substances are operationally defined, rather than chemically defined. This operational definition invites the inclusion of artifacts, confounding the measurements (de Nobili et al., 1988; Iglesias-Jiménez et al., 1992; Miikki et al., 1994; Adani et al., 1995; Veeken et al., 2000). The lack of a standardized method for determining humic substances complicates matters further. Additionally, the literature is unclear/inconsistent as to the extent of humification that occurs in the thermophilic stage compared to the mesophilic stage during composting (Riffaldi et al., 1986; Chefetz et al., 1996; Hsu et al., 1999; Veeken et al., 2000).

2.2.4.3 Nutrient Losses

During the composting process, the concentrations of nutrients may change due to losses via volatilization or leachate runoff and chemical transformations. In the present study, certain nutrients were of interest. Carbon is important since it is a source of energy and biomass synthesis for microbes; nitrogen and phosphorus are primary nutrients for microbes and plants. Nitrogen and phosphorus are also of concern since excessive application rates of soil amendments can cause eutrophication of water sources by the leaching of those nutrients through the soil column or by transport via runoff into surface waters (Edwards et al., 1992 Hunt et al., 1995). Excessive levels of nitrates (> 10 mg/l) in drinking water are also health hazards. The phyto-micronutrients, zinc and copper, are found in relatively high levels in animal manures, especially in swine wastes, due to their use as growth promoters and in antibiotics (Cromwell, 2001). Long term application of animal manures in North Carolina and elsewhere

have resulted in soils with high levels of these nutrients, which is of concern as copper and zinc can be toxic to both plants and grazing animals (Chang et al., 1992; National Research Council, 2005).

Loss of nutrients (via volatilization and leachate losses), which can be considerable, is an inevitable consequence of composting (Martins et al., 1992**;** Warman et al., 1996; Eghball et al., 1997; Tiquia et al., 2002); this is especially true for "windrow" composting (Parkinson et al, 2004). Carbon is lost primarily as CO_2 via microbial respiration; soluble carbon is also lost in leachate (Hsu et al., 1999; Huang et al., 2004). Nitrogen is primarily lost via volatilization of nitrogenous gases (eg, NH_3, N_2, N_xO_y) (Martins et al., 1992; Eghball et al., 1997), ammonia being the primary form (Witter et al., 1988; Martins et al., 1992); organic and inorganic nitrogen can be lost in leachate (Ott et al., 1983; Martins et al., 1992; Nienaber et al., 1994; Eghball et al., 1997). Greenhouse gases, nitrous oxide and methane, are also emitted during the composting process (Hellmann et al., 1997; Hao et al., 2004). The primary mechanism of loss for other nutrients (phosphorus, copper and zinc) is also via leachate runoff (Ott, 1983; Nienaber et al., 1994), as these nutrients are not volatile. However, compared to the other nutrients, carbon and nitrogen incur the greatest losses. In general, the longer the composting time, the greater is the tendency for nutrient losses.

2.2.4.4 Organic Acids

Phytotoxic substances are released during organic matter decomposition (Patrick et al., 1963) of which organic acids are a major component (Toussoun et al., 1968; Manios et al., 1989; Saviozzi et al., 1995). Numerous fatty and phenolic acids have been shown to be phytotoxic (Marambe et al, 1992; Marambe et al, 1993; Shiralipour et at., 1997; Ozores-Hampton et al., 1999). As decomposition progresses, the levels of these phytotoxins tend to decrease (Kuwatsuka et al., 1973; Saviozzi et al., 1995), since the organic acids, which are sources of labile carbon, are metabolized; low molecular weight acids may also volatilize (Paul et al., 1989). Plant bioassays have indicated that extracts from stable composts are less phytotoxic than unstable ones (Zucconi et al., 1981a & b; Chanyasak et al., 1983a; Wong et al., 1985b; Ozores-Hampton et al., 1999), with the hypothesis that the decrease in phytotoxicity was due to the reduction in organic acids.

2.3 Stability and Maturity

2.3.1 General

The terms maturity and stability are used to qualitatively characterize the state of compost during the composting process. These terms have often been used interchangeably, due to the inexactness of the definitions. This author will use *maturity* to refer to the degree of humification and *stability* to refer to the level of microbiological activity (Benito et al., 2005). Uses by other referenced authors are not necessarily the same, nor consistent.

An unstable product indicates that the microbial activity is sufficiently high that use of the product will incur deleterious consequences. Since compost has traditionally been used agriculturally, this infers that plant growth will be negatively affected. As both plants and microbes utilize oxygen for respiration and nitrogen for biosynthesis, high microbial activity can result in low availability of oxygen and nitrogen for plants. High microbial activity also implies the presence of organic phytotoxins or their inevitable release during catabolism of OM. Maturity is a relative term, the reference usually being soil humus or peat.

Quantitating stability and maturity of compost has been elusive, at least on a universal scale. There are neither unanimously accepted definitions nor measurement standards for those terms. Numerous investigations have been concerned with quantitating and optimizing the amount of stability, but there is a paucity of data indicating the degree of maturity that is desirable or necessary. The latter arena is wide open for further investigation.

The degree of humification was not determined for the composts in the present investigation, and therefore, only stability parameters will be discussed. Indices for determining compost stability can be categorized as chemical, biochemical, biological and microbiological, and the literature on them is extensive. A fundamental criterion for a stability index is that the characteristic must either decrease or increase as composting time increases, as a quality that remains invariant over time would not be indicative of transformations within the composting mass. The index must also approach stability with time to reflect a stable microbial community, and thus a stable compost product.

A literature review on stability indices is beyond the scope of the present paper. However, review articles by Iglesias-Jiménez et al. (1989) and Mathur et al. (1993) will provide a more complete picture of stability indices. While not review articles, Forster et al. (1993), Bernal

et al. (1998b) and Goyal et al. (2005) used a variety of indices to assess stability of different compost feedstocks. There is no one index for defining stability (Riffaldi et al., 1986; Chefetz et al., 1996). This review will focus on those potential indices that have relevance to the present study, and include chemical and physical characteristics of the compost, as well as thermodynamic properties, the latter being discussed in Section 2.5.

2.3.2 Stability Indices

2.3.2.1 Organic Matter

During composting, as decomposition progresses organic matter (OM) content decreases, with the consequence of reduced microbial activity, which is an indication of compost stabilization. Organic matter plus ash content is equivalent to total mass, so either one can be used as an index for stability. These indices have been used in conjunction with other ones to establish stability (Ouatmane et al., 2000).

2.3.2.2 Carbon and Nitrogen

The C:N ratio has traditionally been used as an indicator of stability. As composting progresses, this ratio tends to decrease, a stable ratio indicating a stable product. However, for a compost mixture with a low initial C:N ratio, the final C:N ratio may not be indicative of adequately decomposed compost (Eiland et al., 2001a; Huang et al., 2004). The C:N*f* /C:N*i* ratio, where f indicates final and i indicates initial C:N ratios, was proposed as an alternative to the C:N ratio (Morel et al., 1987), although no numerical values were provided. Iglesias-Jiménez et al. (1989) reviewed the literature and concluded that this ratio might be a useful complementary indicator.

2.3.2.3 Ammonium and Nitrate

With an optimum C:N ratio, little excess ammonium (determined as total ammoniacal nitrogen, TAN) is produced during OM decomposition, and thus little nitrate is formed. With increasing decomposition, the C:N ratio decreases, more TAN is produced and consequently more nitrate is formed. However, during the thermophilic phase of composting, little nitrification occurs since temperatures greater than 40^{O}C inhibit nitrifying bacteria (Paul et al.,

1996). As decomposition progresses, the microbial substrate diminishes and microbial activity decreases, resulting in lower production of ammonium. Ammonia volatilization also contributes to reductions in total ammoniacal nitrogen levels. As the compost temperatures decrease to below thermophilic levels, the activity of the nitrifying bacteria increases. As maturation continues, and aerobiosis is maintained, the nitrate concentrations tend to increase, with leachate loss contributing to lower nitrate levels. Stable TAN, and nitrate (NO_3-) concentrations have been used as indicators of compost stability (Riffaldi et al., 1986; Zucconi et al., 1987; Inbar et al., 1993), and the TAN/NO_3- ratio has also been proposed as an index for stability (Bernal et al., 1998b).

2.3.2.4 Organic Acids

Organic acids are released during OM decomposition (Toussoun et al., 1968; Manios et al., 1989; Saviozzi et al., 1995; Baziramakenga et al., 1998). As decomposition progresses, the levels of these acids tend to decrease (Kuwatsuka et al., 1973; Saviozzi et al., 1995; Avnimelech et al., 1996; Baziramakenga et al., 1998), primarily through metabolic decomposition and volatilization. Stable, low concentrations of organic acids and phenols have been proposed as indicators of stability (Keeling et al., 1994a; Liao et al., 1994). Phenol content, as a percentage of OM, has also been proposed as a stability index (Hartenstein, 1981; Riffaldi et al., 1986).

2.3.2.5 pH and Electrical Conductivity

During composting, pH may fluctuate due to a variety of mechanisms. The release of organic acids and carbon dioxide may result in a drop in pH during the early stages of decomposition (Inbar et al., 1993; Nakasaki et al., 1993), while increases in pH have been attributed to the volatilization of ammonia and volatilization and microbial decomposition of organic acids (Paul et al., 1989). Decreases of pH in the latter stages of composting may be due to nitrification. All of the aforementioned processes occur over a wide range of pH values since microbial activity has been reported to be optimum over a wide range, from 5.6 to 8.4 (Jeris et al, 1978; Nakasaki et al., 1993). Although a specific pH range has not been associated with duration of composting since decomposition occurs over such a wide range of pH values, a stable pH has been proffered as an indication of stability (Inbar et al., 1993; Avnimelech et al., 1996).

Electrical conductivity (EC) is dependent upon soluble ions in solution. Contributors include both inorganic and organic species. Increases in EC may correspond with the release of inorganic and organic acids and inorganic ions as decomposition progresses (Inbar et al., 1993). Ionic losses in leachate (Eghball et al., 1997) can buffer elevations in EC, although the loss of mass during composting tends to concentrate ions so that EC may apparently increase. As with pH, a stable EC may indicate stability (Avnimelech et al., 1996; Wang et al., 2004). Although pH and EC have been proposed as indices for stability, they are dependent upon the feedstock and composting conditions, and should be used in conjunction with other indices (Inbar et al., 1993; Avnimelech et al., 1996; Wang et al., 2004).

2.4 Uncomposted/Unstable Amendments

Stable composts, made from agricultural, industrial or municipal solid wastes, have been shown to enhance soil properties and improve crop yields (Giusquiani et al., 1995: Serra-Wittling et al., 1996; Tripepi et al., 1996). Soils amended with uncomposted/unstable organics may result in reduced seed germination and plant yields, due to 1) low redox potential caused by high microbial activity (Miller et al., 1985; Katayama et al., 1987), 2) high carbon/nitrogen ratios, resulting in nitrogen immobilization (Edwards et al., 1993), or 3) high levels of soluble salts (Chanyasak et al., 1983b; Iannotti et al., 1994; Barker, 2001), or metals (Wong et al., 1983; Tiquia et al., 1998) which can be phytotoxic. The presence of transient phytotoxins, especially during the early stages of organic decomposition, can also contribute to the deleterious effects. Ammonia (Wong et al., 1983; Katayama et al., 1985; Barker, 2001), ethylene oxide (Wong et al., 1983), fatty acids (Chanyasak et al., 1983b; Manios et al., 1989; Marambe et al., 1993), and phenols (Marambe et al., 1992) have been identified as phytotoxic substances.

The composting process is a relatively protracted process, especially the "windrow" method. In an attempt to minimize nutrient losses and economic costs (by reducing composting time), less composted feedstocks have been studied for use as soil amendments. Although plants grown in unstable-compost amended soils have exhibited inhibited growth and lower yields (Chanyasak et al., 1983a & b; Keeling et al., 1994b**;** Campbell et al., 1995; Chefetz et al., 1996; Keeling et al., 2003), these deleterious effects are not consistent, being dependent upon soil, plant species and feedstock characteristics (Baca et al., 1990; Chong et al., 1994; Maynard, 1997).

Other investigators have reported that less composted feedstocks are at least as effective as soil amendments as their more composted counterparts (Baca et al., 1990; Keeling et al., 1995; Chefetz et al., 1996; Bernal et al., 1998a; Sela et al., 1998). Thus, although the conventional objective for composting has been to obtain a stable/mature product, fully stable compost may not be necessary for all agricultural applications.

2.5 Calorimetry

Combustion calorimetry is the conventional method for determining the energy content of foodstuff and fuels. This technique measures that change in heat energy that occurs when organic matter is combusted (oxidized). Thermodynamically, this energy change or *enthalpy* change is a measure of change in the strength and number of atomic bonds that are broken and reformed (heats of formation) during the oxidation process.

The metabolism of food, a decomposition process, and combustion are both oxidative processes. However, decomposition is a microbially mediated process, while combustion is a purely chemical one. Calorimetric values of bio-oxidation are lower than those values of combustion of the same materials (Paine, 1971; Lovrien, 1980; Reh et al., 1991). Chemical combustion assumes the complete oxidation of organic matter and carbonaceous matter (ie., end products of CO_2, H_2O, N_2). Bio-oxidation is a function of the efficiency of the microbial process, and the primary final products of nitrogen oxidation are ammonia and nitrate, not nitrogen gas.

There is an inverse relationship between calorific values and organic matter content or time of OM degradation (Kucera et al., 1967; Rsyzkowski et al., 1975; Bieńkowski, 1981; Zanoni et al., 1982; Hegazy, 1998). However, when caloric values are calculated on an organic matter basis, the values are related to the recalcitrance of the OM. As decomposition progresses, the labile organic components are consumed prior to the more recalcitrant ones, and labile forms are also transformed into more refractory entities (Wilson, 1985; Lähdesmäki et al., 1988; Collins et al., 1990; Bernal et al., 1992; Tachimoto et al., 1995; Adani et al., 1995; Hsu et al., 1999). Kucera et al. (1967) and Reh et al. (1990) hypothesized that as the recalcitrance to microbial degradation increases, calorific values increase, with a consequent shift to higher calorific values as decomposition progresses (Bocock, 1964; Kucera et al., 1967; Lähdesmäki et al., 1988). Calorific

values of organics have the following general descending hierarchy: lipids>proteins>carbohydrates [complex > simple] (Kharasch, 1929; Kleiber, 1961; Gary et al., 1995; Rhén, 2004); lignin > cellulose=hemicellulose (Gary et al., 1995; Rhén, 2004). However, each category of OM (in the former grouping) comprises a wide range of molecular structures and weights, and consequently, the calorific values and biodegradabilities vary widely within each category. Therefore, the relative calorific levels only generally correspond to the relative categories of biodegradability: lignin > cellulose > [lipids, proteins, carbohydrates] (Haider, 1992; Epstein, 1997).

Since OM provides the energy for microbial activity, which is integral for edaphic changes and plant growth, the energy content of the OM may indicate the potential to improve soil quality and plant growth. Kirchmann et al. (1990) designated composted manure/straw mixtures as being energy poor and fresh manure/straw mixtures as being energy rich, when describing the nitrogen availability of the two mixtures. Vincelas-Apka et al. (1994) used calorimetry to characterize the composting process, although there have been no studies relating calorific levels of compost to growth media and plant response.

2.6 Soil Physical Properties

2.6.1 Overview

Organic amendments have been reported to improve soil physical properties (Somani et al., 1975; Mbagwu, 1989; Tester, 1990; Shiralipour et al., 1992; Aggelides et al., 2000; Chambers et al., 2002), and consequently enhance plant growth. Soils that promote plant growth are those that are readily penetrated by roots, have the appropriate water holding capacity to allow adequate water for plants and allow adequate air and water transmission. Bulk density, soil impedance, water holding capacity and water aggregate stability are physical properties commonly used to quantify soil quality. In the present study these four soil characteristics were used to assess the effectiveness of soil amendments.

2.6.2 Density, Water Holding Capacity and Impedance

OM tends to reduce bulk density by dilution, as the less dense organics are mixed with the more compact soil mineral fraction, and also by inducing changes in the soil aggregate

structure that increase porosity/pore size distribution (Pagliai et al., 1981). Khaleel et al. (1981) reported an inverse relationship between organic matter additions and bulk density.

Soil impedance is a measure of the ability of the soil to resist penetration. This characteristic is related to bulk density and is often used to characterize soil compaction. As bulk density increases (eg, compaction), the number and size of pores decreases. Consequently, it is more difficult for roots to penetrate and water and air transmission is also impeded. While bulk density is a requisite measurement for soil characterization, the use of penetrometers is not extensive due to the dependence of the measurements on several factors, such as soil water content, soil texture, and lack of standard design of the cone penetrometer and standardized procedures (Hillel, 1982; Motavalli et al., 2003). However, the ease and speed of measurements has made the penetrometer a useful tool in indicating the root growth potential in soils (Thompson et al., 1987; Vepraskas et al., 1989).

The water holding capacity (WHC) is important as it describes the water retention and drainage capability of soil. Two points are often referenced in characterizing water holding capacity: field capacity and wilting point. Field capacity is the amount of water soil can retain with only gravity draining the water and is often determined at a tension of –0.01 MPa. Wilting point is the point designated where plant roots can no longer imbibe water from the soil and is determined at a tension of –1.5 MPa, so that only the smallest pores are able to retain water. Organic matter increases soil water retention due to the high active surface area and its enhancement of pore structure (Khaleel et al., 1981; Pagliai et al., 1981; Brady, 1990). Khaleel et al. (1981) reported a positive correlation between organic matter additions and water holding capacity.

2.6.3 Water Aggregate Stability

2.6.3.1 Aggregate Formation

The stability of soil aggregates is necessary for the maintenance of soil integrity, since the stability affects the movement and storage of water, aeration, erosion, biological activity, and crop growth (Amézketa, 1999). In general, hydraulic conductivity, infiltration rate, air diffusivity, surface drainage and ease of root penetration will increase with increasing aggregation and stability (Clapp et al., 1986); resistance to erosion also improves (Luk, 1973; Clapp et al., 1986).

Aggregate stability is a measure of the resistance of soil aggregates to breakdown. Wet (or water) aggregate stability (WAS) is measured by wet sieving techniques and also includes the ability to resist the stresses due to unequal swelling or entrapped air when wetted (slaking), but usually excludes the erosive forces of water impaction which only occur at the soil surface.

There are two theories of aggregate formation. One theory proposes that macro-aggregates (> 250um) are formed from microaggregates (< 250 um) (Tisdall et al., 1982). Oades et al. (1991) proposed that microaggregates are formed from the disintegration of macro-aggregates, which are formed by the compaction and restraint of roots and hyphae of fungi. Both theories have merit and most likely act in concert.

Aggregation/stability involves a combination of both organic and inorganic chemical factors, plus the physically stabilizing effects of fungal and root hyphae (Stotzky, 1986; Tisdall, 1996; Robert et al., 1992). The inorganic factors include those entities that promote aggregation such as clays, polyvalent cations (eg, Fe, Al), oxide and hydroxides of Fe, the carbonates of Al, Ca and Mg and gypsum ($CaSO_4 \cdot H_2O$), with the metal moieties acting as cation bridges between negatively charged organic/inorganic particles (Tisdall et al., 1982; McBride, 1994). Clays, with their relatively high surface areas, promote particle aggregation/stability more than the coarser fractions of soils (Kemper et al, 1986; Clapp et al., 1986.).

Organic matter has been associated with enhanced soil aggregation and stability (Greenland et al., 1975; Haynes et al., 1990; Chambers et al., 2002), and constitutes a disparate mixture of organic moieties that range from readily biodegradable (such as carbohydrates, and proteins) to refractory (such humic acids, lignin and tannins), all of which contribute to particle aggregation and stability. In the surface layer of agricultural soils, where most of the organic matter exists, organic matter plays a pivotal role in binding aggregates (Tisdall et al., 1982; Oades et al., 1991).

Particles of OM may act as nuclei for aggregate formation (Oades et al., 1991; Beare et al., 1994; Plante et al., 2002), while different forms of OM act as promoters of aggregation and stability (Tisdall et al., 1982; Oades et al., 1991). Tisdall et al. (1982) defined three categories of organic matter that contribute to aggregation/stability: transient, temporary and recalcitrant. The transient binding agents, which are readily biodegradable, are carbohydrates (polysaccharides) and polyuronides (Rennie, 1954; Tisdall et al., 1982; Oades, 1984; Lax et al., 1993; Tisdall, 1996).

Exudates from soil microbes, fungi, plant roots, and constituents of decomposing organic matter are sources for the carbohydrates (Rennie et al., 1954; Oades, 1978; Lynch, 1981; Haynes et al., 1997). OM stimulates microbial proliferation and root growth, which increases production of these extracellular secretions. The temporary organic matter are the plant roots and hyphae of fungi that physically entwine particles (Tisdall et al., 1982; Robert et al., 1992; Davet, 2004). Recalcitrant organic matter, such as humified OM may act as long term binding agents (Chaney et al., 1986; Fortún et al., 1990), as do the inorganic moieties (Muneer et al., 1989b; Oades et al., 1991), discussed previously.

Organic matter that is associated with soil particles may reduce aggregate slaking by its ability to reduce water entry into pores (Haynes et al., 1990; Sullivan et al., 1990; Zaher et al., 2005). This may occur due to the hydrophobic character of certain organic matter fractions and the physical occlusion of pores.

Aggregation and stability are not necessarily synonymous, as size per se, does not confer stability (Dormaar, 1983; Angers et al., 1988; Sort et al., 1999; León-González et al., 2000). Stability depends upon moieties forming the aggregates and the ability of the bond to maintain its integrity (ie, transient vs. recalcitrant OM).

2.6.3.2 Soil Factors

2.6.3.2.1 Abiotic Soil Factors

Texture and mineralogy are important factors in how a soil responds to organic amendments. Clays are mineral particles with equivalent spherical diameters of less than 2 μm. The particles have a net negative charge, and the high specific surface areas make them more reactive than the silt or sand components of soil. Clapp et al. (1986) reported that sewage sludge was more efficacious in promoting aggregate stability in fine-textured soils.

Clays with a 2:1 layered molecular structure have greater surface areas than clays with 1:1 layered structures, due to interlayer reactivity. Higher surface area plus greater isomorphous substitution in 2:1 clays translates into greater cation exchange capacity (CEC), which is a quantitative measure of the reactivity of negatively charged surfaces. The 1:1 clays have a CEC range of 1-15 cmoles/kg, 2:1 clays have a CEC range of 10-150 cmoles/kg, and those 2:1 clays which are able to absorb water interstitially, and thus expand, have the largest CECs of 70-150

cmoles/kg (McBride, 1994). Differences in CEC between different types of clays affect interactions with organic and inorganic moieties, and consequently affect soil physical properties.

Six et al. (2000a & b) concluded that a soil containing high concentrations of kaolinite and oxides of iron and aluminum reacted less with OM than soils dominated by high CEC components (2:1 mineralogy). Reductions of soil organic matter resulted in a smaller decrease of wet aggregate stability in soils dominated by the oxides and 1:1 minerals than in soils dominated by 2:1 minerals (Six et al., 2000b). Six et al. (2000a) reported that in soils dominated by 2:1 clay mineralogy, the carbon content of macro-aggregates was greater than in micro-aggregates. In the soil with large amounts of kaolinite and oxides, aggregate carbon did not increase with increasing aggregate size. The authors concluded that the results supported an aggregate hierarchy theory in which micro-aggregates are bound together into macro-aggregates by organic binding agents in 2:1 clay-dominated soils, but less so in soils with low CEC.

These latter conclusions agree with the earlier work of Oades et al. (1991), who reported that an oxisol soil, dominated by kaolinite and iron oxide, did not exhibit the type of disaggregation as those soils containing higher CEC clays, illite (2:1 non-swelling clay)/kaolinite mix or smectite (2:1 swelling clay). Besides being more resistant to breakdown, the macro-aggregates in the oxisol disaggregated into less than 20 μm particles in contrast to the other soils where the macro-aggregates broke down into smaller units in a more stepwise fashion. A variety of sizes of aggregates would indicate different bonding mechanisms associated with the various types of organic structures that are involved in the aggregation process, while one with a complete breakdown, such as occurred with the oxisol, would indicate primarily one type of aggregation mechanism. The authors concluded that OM did not play a pivotal role in the aggregation process in the oxisol.

Oxides/hydroxides of iron and aluminum have relatively low cation exchange capacities (~3 cmol/kg for crystalline forms, Brady, 1990) and tend to be positively charged at normal soil pH (McBride, 1994), promoting particle-particle flocculation of the negatively charged clay particles via cation bridging (El-Swaify et al., 1975; McBride, 1994). The combination of low CEC clays and the presence of iron or aluminum oxides would tend to reduce the effects of added OM. Particle flocculation is also enhanced by other multivalent cations such as calcium (van Olphen, 1977), and lime has been reported to increase WAS (Rennie et al., 1954). However,

calcium may also mute the aggregating potential of OM, for example, in calcareous soils (Stefanson, 1968 & 1971; Guidi et al., 1988).

2.6.3.2.2 Plants

The presence of plant roots increase aggregate stability compared to soils that lack them (Monroe et al., 1987; Haynes et al., 1997). This is due to both the roots themselves and associated microbial community (Caravaca et al., 2002), as plants promote microbial activity in soil (Schnürer et al., 1985). Secretions from plant roots act as binders between particles, and fungal hyphae associated with the roots also physically entwine soil particles to enhance aggregate stability (Oades, 1978; Oades et al., 1991; Robert et al., 1992; Davet, 2004). However, plant growth does not necessarily increase aggregate stability, the magnitude and effectiveness being dependent upon the plant species/cropping system (Monroe et al., 1987; Angers et al., 1988; Haynes et al., 1997).

2.6.3.3 Soil Amendments

2.6.3.3.1 Composted Organic Matter

The addition of organic matter to soil enhances water stability of soil aggregates. Composting OM reduces OM nutrient concentrations and transforms OM labile nutrients into more recalcitrant forms. Subsequently, the effects of composted OM on soil aggregation appear to be dependent on the duration of composting. Martin (1942) and Sela et al. (1998) reported that OM composted for short periods of time induced the greatest WAS compared to less or more composted OM. Martin (1942) hypothesized that this phenomenon was due to increasing amounts of labile nutrients produced during short term composting. Other investigators have confirmed that decreases in nutrients during composting are not necessarily monotonic (Hsu et al., 1999; Sánchez–Monedero et al., 1999; García-Gómez et al., 2005). Sela et al. (1998) reported that short term (7-14 day) composting induced the greatest WAS, although the authors did not interpret this "spike" as a perturbation. Rather, a perturbation was caused by the uncomposted feedstock. The authors hypothesized that the uncomposted OM induced high microbial activity causing low oxygen conditions (due to the larger pool of bioavailable nutrients), which resulted in low carbohydrate production, contributing to lower WAS (Avnimelech et al., 1988). Further

composting led to reductions in WAS, presumably as the nutrient pool diminished. However, an alternate explanation or contributing factor could be the fact that the decrease in nutrients during composting was not monotonic, and the "spike" in WAS after short term composting was due to a temporary increase in labile nutrients.

2.6.3.3.2 Fertilizer

The effects of nitrogen containing fertilizers on aggregate stability are inconclusive. Skinner (1979) added a nitrogen rich organic (peptone - water soluble byproducts of protein hydrolysis) to soil and found that soil particle aggregation tended to decrease compared to unfertilized soil, after a 14 day incubation. Apparently, excess nitrogen created an imbalance and insufficiency of soil carbon causing the microbes to catabolize polysaccharides, which are binding agents, and also readily available sources of carbon (Diaz-Burgos et al., 1993), resulting in the decrease in WAS (Rennie et al., 1954 ; Skinner, 1979). In long term field studies (at least 10 years), aggregate stability has been reported to increase due to nitrogen containing mineral fertilizers compared to controls not receiving nitrogen (Ibrahim et al., 1999; Albiach et al., 2001b). However, in the study of Albiach et al. (2001b) the differences were not statistically significant, and no statistical comparisons were made in the investigation of Ibrahim et al. (1999) due to the lack of replication.

Increased microbial activity and plant growth tend to enhance WAS, and microbial activity and plant growth are positively correlated (Perfect et al., 1990a; Caravaca et al., 2002). Reductions in microbial activity after additions of nitrogen containing fertilizers have been reported on fallow fields after 1-3 years of annual applications (Kowalenko et al., 1978), the declines being attributed to reductions in pH due to nitrification (Kowalenko et al., 1978) and increased salt concentrations causing increased osmotic potential (Johnson et al., 1963). However, on cultivated fields the declines in microbial activity have not been confirmed, and have tended to be the opposite. In a three year study, Petersen et al. (2003) reported variable results, depending upon the soil characteristics and specific indicator of microbial activity. In a long term study, Kautz et al. (2004) reported that biochemical indicators of microbial activity and size of the microbial communities did not statistically change when soil was amended with nitrogen containing fertilizer, compared to unfertilized soil.

Kirchner et al. (1993) and Albiach et al. (2001b) reported that microbial activity in fertilized soils increased compared to non-nitrogen fertilized soils in a cultivated field, although the differences were generally not statistically significant. Kirchner et al. (1993) concluded that microbial activity was at least partially dependent upon plant growth, since roots provide nutrients for microbes (Davet, 2004), although previous harvest residues may also have contributed to the confounding. Since fertilizers increase plant growth (Moraghan et al., 1984a & b; Petersen et al., 2003), it may not be possible to separate the effects of nitrogen fertilizer on WAS from plant growth on cultivated fields.

2.6.3.4 Climatic Factors

2.6.3.4.1 Seasonal Fluctuations

Wet aggregate stability fluctuates during the year as climatic conditions change and is also affected by the plant species/cropping system (Rennie, 1954; Webber, 1965; Perfect et al., 1990a; Beare et al., 1993). Seasonal fluctuations have been found to be maxima in the summer and minima in the winter (Alderfer, 1946; Wilson et al., 1947; Rennie, 1954; Stefanson, 1971; Bullock et al., 1988; Guidi et al., 1988), although statistically significant differences have not necessarily been reported.

Climatic conditions affect soil microbial activity and plant growth. In the warmer periods, microbial communities proliferate and in the colder seasons, they decline (Lynch et al., 1980; Saviozzi et al., 1993), as does plant growth. These trends parallel the temporal water stable aggregate fluctuations. Microbial population trends also follow the aggregate stability trends during a growing season (Guidi et al., 1988).

2.6.3.4.2 Precipitation

Moisture content of the soil effects microbial activity, with optimum activity being at about field capacity, approximately -0.03 - 0.01 MPa (Robert et al., 1992; Paul, 1996; Agehara et al., 2005). Low rainfall can reduce microbial activity and contribute to the reduced effectiveness of organic matter in promoting aggregate stability (Alderfer, 1946; León-González et al., 2000), while a high accumulation of precipitation can cause low oxygen conditions, also diminishing microbial activity and reducing aggregate stability (Avnimelech et al., 1988).

2.7 Nutrient Dynamics

2.7.1 Microbial Community

Additions of OM to soil stimulate microbial activity as the OM is a source of nutrients. For microbes, carbon is required in the greatest amounts followed by nitrogen, phosphorus and sulfur; other nutrients, such as copper and zinc are also required, but at lower levels. The greater the amount of labile nutrients in the OM, especially carbon and nitrogen, the greater the stimulatory effect (Tester et al., 1977; Nishio et al., 1980; Saviozzi et al., 1993; Pascual et al., 1998). Soil organic matter is comprised of significant amounts of recalcitrant forms of OM, and therefore exogenous additions of OM to soil will generally stimulate soil microbial proliferation.

The OM (non-living) will directly stimulate saprobic, heterotrophic microbes. Saprobes decompose dead organic matter. Heterotrophs (eg, many bacteria, fungi) obtain carbon from organic sources for use as an energy source and for biosynthesis. As soil within the plow layer (approximately 15-20 cm depth) is primarily an aerobic environment, microbes that utilize aerobic respiration for metabolic function will proliferate. However, other microbial populations are indirectly stimulated by OM additions. Catabolism of organic matter produces carbon dioxide. Lithotrophs metabolize inorganic compounds for energy (ie, reduced compounds of nitrogen, iron and sulfur). Chemolithotrophs use carbon dioxide as a source of carbon for biosynthesis and inorganic compounds as an energy source. Similarly, chemoorganotrophs utilize inorganic compounds for energy, but organic carbon sources for biosynthesis. Heterotrophic, predatory microorganisms, such as protozoa, also proliferate as their prey multiply.

2.7.2 Carbon and Nitrogen

Carbon and nitrogen are required in large amounts in all living systems. Carbon and nitrogen compounds are used for energy production and biosynthesis. In soil, carbon is either in an organic or inorganic form (CO_2, HCO_3- or CO_3-). Nitrogen is found primarily in the organic form (eg, amino acids) in soil, although ammoniacal and nitrate nitrogen are the most stable inorganic forms.

In aerobic environments, microbial respiration produces carbon dioxide, water and energy as endproducts, with carbon being oxidized (organic to inorganic transformation) and

oxygen reduced (molecular oxygen to water transformation). Heterotrophic microorganisms convert exogenous nitrogen moieties into organic microbial biomass or byproducts (assimilation).

During metabolism, microbes utilize carbon and nitrogen in the same ratio as their cellular biomass. A carbon:nitrogen ratio of approximately 20-30 has been found to optimize the efficiency of microbial metabolism, although the bioavailability of the nutrients is critical (section 2.2.4.1). Above this optimal threshold, nitrogen is deficient resulting in nitrogen assimilation. Below the threshold, nitrogen is in excess and ammonium is produced (a mineralization process). As decomposition of OM progresses, the C:N ratio decreases due to nutrient losses (section 2.2.4.3), resulting in increasing amounts of ammonium. Nitrifying bacteria are chemolithotrophs and use reduced nitrogen as an energy source. *Nitrosomonas* oxidize ammonium to nitrite and *Nitrobacter* oxidize nitrite to nitrate. The nitrogen transformations in the nitrification process are shown in equation 2-1:

$$NH_3 \rightarrow NH_2OH \rightarrow [N_2O \rightarrow NO] \rightarrow NO_2\text{-} \rightarrow NO_3\text{-} \qquad (2\text{-}1)$$

During nitrification, nitrogen losses due to nitrous and nitric oxide have been measured (Paul et al., 1993; Skiba et al., 1993). Ammoniacal nitrogen concentrations can decrease due to volatilization of ammonia, which is promoted by alkaline conditions.

Low oxygen conditions in soil can be caused by high microbial activity or high moisture content. Aerobic respiration requires oxygen (terminal electron acceptor). When the availability of oxygen becomes insufficient due to slow diffusion of oxygen (high moisture) or high respiration (high aerobic microbial proliferation), aerobic microbial activity diminishes and the anaerobic microbial activity increases. With oxygen being less available as a terminal electron acceptor, other oxidized moieties (eg, oxides of nitrogen, iron and sulfur) are reduced. Nitrate can be reduced via different anaerobic pathways. Denitrification is the mechanism when nitrogen gas (N_2) is produced. Dissimilatory nitrate reduction (DNR) is the pathway for energy needs and ammonium is the final product; if the ammonium is further reduced to organic compounds for biosynthesis, the mechanism is termed assimilatory nitrate reduction (ANR). DNR is promoted by carbon (labile) rich environments, and is therefore not generally

predominant in soil, where denitrification is the dominant mechanism. Carbon rich conditions also enhance ANR, although high concentrations of ammonium inhibit this pathway as assimilating extant ammonium requires less energy than reducing nitrate. The process of denitrification (eq. 2-2) indicates the nitrogen transformations) results in emission of nitrous and nitric oxides and nitrogen gas.

$$NO_3 \rightarrow NO_2 \rightarrow NO \rightarrow N_2O \rightarrow N_2 \qquad (2\text{-}2)$$

Ammonium and nitrate are very transient in the soil due to the utilization of both by microbes and plants. Since ammonium is positively charged, it is not very mobile in soil, being readily adsorbed to the negatively charged inorganic clay fraction of the soil or negatively charged ligands of organic matter, although it is also generally readily exchangeable with other cations. However, non-exchangeable adsorption (fixation) does occur with clays having a 2:1 layered structure, especially vermiculite. Conversely, nitrate is very mobile in soil, being negatively charged. Ammoniacal nitrogen can be replenished in the soil via nitrogen fixation, the conversion (reduction) of nitrogen gas to ammonium.

2.7.3 Phosphorus and Sulfur

2.7.3.1 Phosphorus

Phosphorus exists in both organic and inorganic forms in soil, with up to approximately 75% being organic (Stevenson, 1994). Phosphorus primarily exists as orthophosphate (PO_4) and at normal soil pH range of 5-8, the orthophosphate structures are primarily $H_2PO_4^-$ and HPO_4^{-2} in solution being organically or inorganically bound. Phosphates often complex with calcium, iron or aluminum, and due to the low solubility of these inorganic complexes relatively low amounts of phosphorus are lost from the soil via leaching; as phosphorus compounds are not volatile, emission losses are negligible. However, phosphate leaching is enhanced by organic matter, which promotes solubilization of phosphates by complexing with metal cations (Stevenson, 1994).

As with nitrate-nitrogen, microbes and plants utilize phosphates. Microbes assimilate or mineralize organic phosphorus depending upon the (total C):(total P) ratio, with the demarcation

being approximately 100:1 to 150:1 (Enwezor, 1976). Above the threshold, phosphorus assimilation predominates and below the threshold net mineralization occurs. Stevenson (1986) indicated that net mineralization occurs when the C:organic P was less than 201 and net assimilation dominated when the ratio was at least 300. Contrary to nitrogen, phosphorus is not generally oxidized or reduced by microorganisms.

2.7.3.2 Sulfur

Sulfur exists in both organic and inorganic forms, although in agricultural soils, inorganic sulfur constitutes less than 25% of the total sulfur, with sulfates being the most common form (Stevenson, 1986). Microbes and plants utilize sulfate (SO_4^{-2}). Mineralization/assimilation dynamics for sulfur are analogous to those of nitrogen: net mineralization dominates for a carbon (C):sulfur (S) ratio of less than 200 and net assimilation occurs for a C:S ratio of greater than 400 (Stevenson, 1986). For soil organic matter, a typical carbon:nitrogen:phosphorus:sulfur ratio is 140:10:1.3:1.3 (Stevenson, 1986).

Sulfur is similar to nitrogen in that it can exist in several oxidation states, and is therefore a source for energy (terminal electron acceptor) and biosynthesis. Reduction of sulfur can occur via two different pathways: transformation into organic sulfur (assimilatory reduction) or hydrogen sulfide (dissimilatory reduction), the latter being analogous to denitrification. The rate of sulfate reduction increases with decreasing redox potential (Eh), the redox potential being positively correlated with oxygen levels. An Eh of approximately 350 mV indicates that a deficit of oxygen exists and consequently alternative oxidized moieties are used as terminal electron acceptors by microbes. Nitrate is reduced at a redox potential of approximately 250 mV and sulfate is reduced at approximately –150 mV (Brady, 1990). Reduction of sulfates can lead to gaseous losses of H_2S and organic gases (eg, CS_2, COS and CH_2SH).

The oxidation (mineralization) of sulfur moieties is primarily a microbially mediated process, and can occur over a wide pH range (~2-9) (Stevenson, 1986). This is in contrast to the analogous nitrification process, which is sensitive to low pH. Lithotrophs (microbes using either light or inorganic carbon compounds for energy and the latter for biosynthesis) and chemoheterotrophs appear to be the important sulfur oxidizing microbes in soil; sulfate reducing microbes are primarily chemoheterotrophs (Stevenson, 1986; Germida et al., 1992).

Being negatively charged, sulfate is easily leached down the soil profile. However, sulfates readily complex with calcium, magnesium, iron and aluminum forming less soluble moieties, which reduce potential leaching.

2.7.4 Copper and Zinc

Copper and zinc are micronutrients for both microbes and plants. The dominant forms of soluble species in soil are Cu^{+2} and Zn^{+2} and $Zn(OH)^{-}$ (Brady, 1990). The mobility of copper and zinc is low due to their ability to readily form less soluble complexes with anionic ligands, such a sulfide (in low oxygen environments), hydroxide and organic matter. As pH increases, the bioavailability of these nutrients tends to decrease. Similar to phosphorus, the low solubility of the aforementioned cation complexes result in relatively low amounts of copper or zinc being lost from the soil via leaching and as copper and zinc compounds are not volatile, emission losses are negligible. Conversely, dissolved organic matter can solubilize these cations, increasing leaching potential (McBride, 1989; Zhou et al. 2001; Karathanasis et al., 2005).

2.8 Research Objectives

1) Co-compost swine waste solids for different lengths of time and characterize the composts thermodynamically and chemically.

2) Use the composts as soil amendments and evaluate their effects on plant growth and chemical and physical characteristics of the soils.

3) Relate compost stability characteristics to the effects on soil properties and plant growth.

4) Determine nitrogen mineralization potentials of the organic amendments in a laboratory incubation study and compare the results to the projected rates, which were hypothesized from the literature.

Chapter 3
Pilot Study

3.1 Background

The pilot study was conducted to "optimize" the ratio of feedstock (swine manure solid) to bulking agent (chipped Paulownia branches and shredded leaves) when using the windrow composting method. True optimization necessitates a closed (controllable) system and consistent characteristics of starting materials. The windrow process is an open system, and ambient conditions can affect the composting process (Parkinson et al., 2004). In addition, the variabilities of the manure and Paulownia trees were unknown. Therefore, this pilot study was not a true optimization.

The primary objective of the optimization was to obtain compost pile temperature profiles that would adhere to Federal Regulations 40 CFR, Part 503 to produce a Class A product with respect to pathogen/vector attraction reduction (Chapter 4.1.2). Carbon and nitrogen concentrations were also monitored to determine nutrient retention.

Previous investigations co-composting swine manure (M) and peanut shells (PS) had indicated that a volume ratio of 3M:2PS (wet weight basis) obtained pile temperatures of at least 55^{O} C. Whereas the peanut shells were relatively rigid, the chipped wood/leaf bulking agent had less composite rigidity. Therefore, it was felt that smaller manure to bulking agent ratios might be warranted in this pilot study. The ratios of swine manure solids (M) to bulking agent (P) selected were: 2M:3P, 1M:1P and 3M:2P, on a wet weight basis.

3.2 Experimental Design

The process objective was to achieve pile temperatures of at least 55^{O} C for 15 days, with a minimum of 5 pile turnings during that time period, per Federal regulation 40 CFR Part 503. The estimated time for the compost pile temperatures to go through a mesophilic-thermophilic-mesophilic temperature cycle was approximately four weeks, since previous studies with manure/peanut shell mixtures indicted it took about three to four weeks.

Three piles were mixed, one pile for each of the selected swine manure solids

(M):bulking agent (P) ratios, abbreviated 2M (2M:3P), 1M (1M:1P) and 3M (3M:2P). The piles were approximately 2 m X 1 m X 1 m. A moisture content of approximately 50% was maintained until the pile temperatures had completed the thermophilic phase (> 45^{O} C). The temperatures were measured at five locations at a depth of approximately 30 cm. Temperatures were monitored almost daily; moisture content was measured less frequently. The piles were processed under sheltered conditions at the Animal and Poultry Waste Management Center at North Carolina State University.

Samples for moisture and chemical analyses were homogenized from about five sampling points within each pile. Total carbon, total nitrogen, total Kjeldahl nitrogen (TKN), nitrate and total ammoniacal nitrogen (TAN) levels were determined. Total carbon and nitrogen were determined by combustion, using a Leco C:N 2000 Autoanalyzer (Leco, St. Joseph, MO). TKN was determined using a persulfate digestion (Schuman et al. 1973) and analyzed per Standard Methods (1998) 4500Norg-B. TAN and nitrate (including nitrite) were extracted with K_2SO_4 (Keeney et al., 1982) and analyzed per Standard Methods (1998) 4500-NH3-G and 4500-NO3-E, respectively.

3.3 Results and Discussion

The durations of the mesophilic-thermophilic-mesophilic temperature cycle were approximately four to six weeks (Figure 3.3-1). All of the pile temperatures exceeded the 55^{O} C minimum, although only the 2M and 1M piles maintained that minimum for at least 15 days; pile 3M sustained the minimum temperature for 14 days. Pile 1M maintained the minimum temperature for the longest time period.

The temperatures of the 2M pile spiked to over 70^{O} C for several days. Optimum microbial degradation of organic matter is deemed to be less than about 65^{O} C (Chang et al., 1967; Ladd et al., 1972, MacGregor et al., 1981; Sikora et al., 1985; Strom, 1985). Also, temperatures that are too high promote the regrowth of pathogenic microbes (Burge et al., 1987).

From temperature considerations, pile 1M was the preferred mixture. In addition, the strategy for manure utilization would be to maximize the use of the waste. The 1M mixture used a greater proportion of manure than 2M. Thus, the 1M mixture was selected as the optimum one.

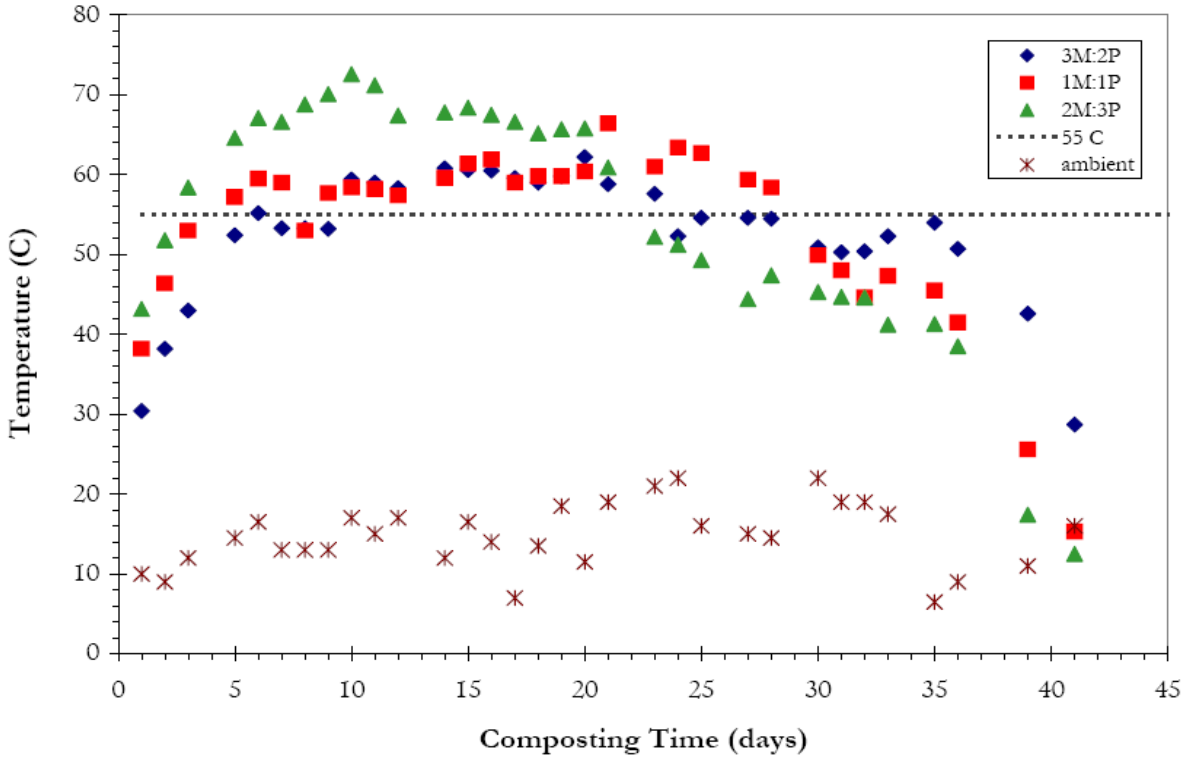


Figure 3.3-1: Pile temperatures at 30 cm depth for composts 3M:2P (◆), 1M:1P (■) and 2M:3P (▲). Ambient temperature (✳) is indicated, as is the 55^{O} C reference temperature (----).

Figures 3.3-2 to 3.3-4 indicate the changes in nitrogen concentration of 1M. Figure 3.3-5 shows the carbon/nitrogen ratio during the composting period. The trends of the temperature profiles and general carbon and nitrogen dynamics will be covered within the context of the results of the main experiment (Chapter 5).

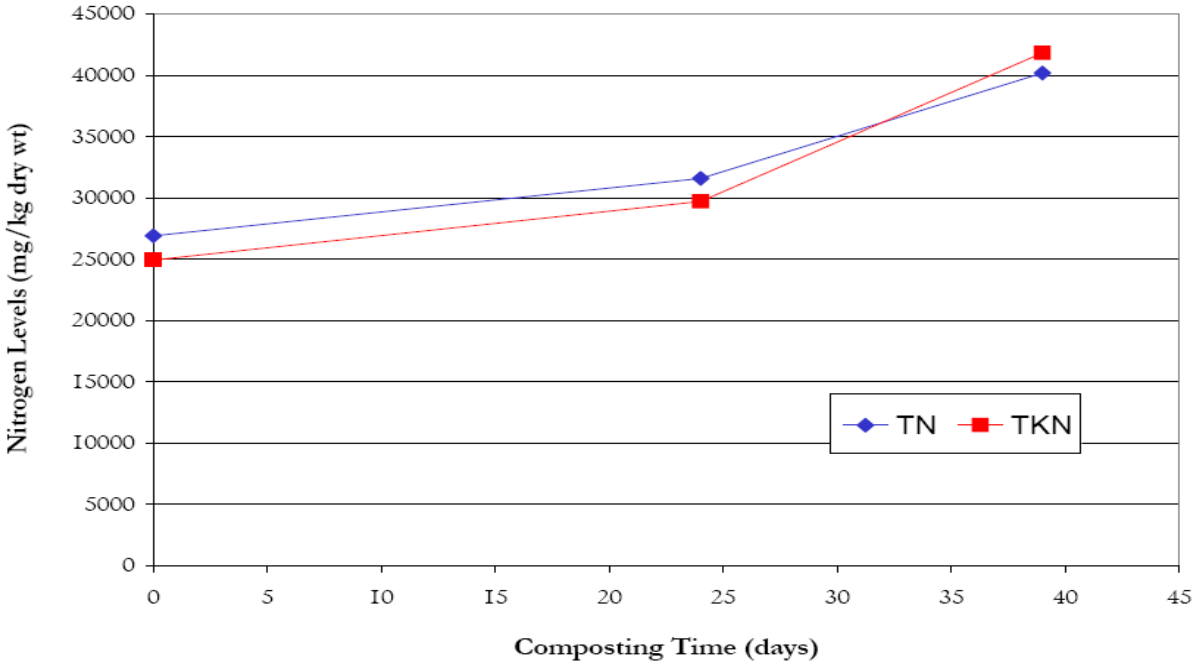


Figure 3.3-2: Total nitrogen (◆) and total Kjeldahl nitrogen (■) levels during composting of 1M

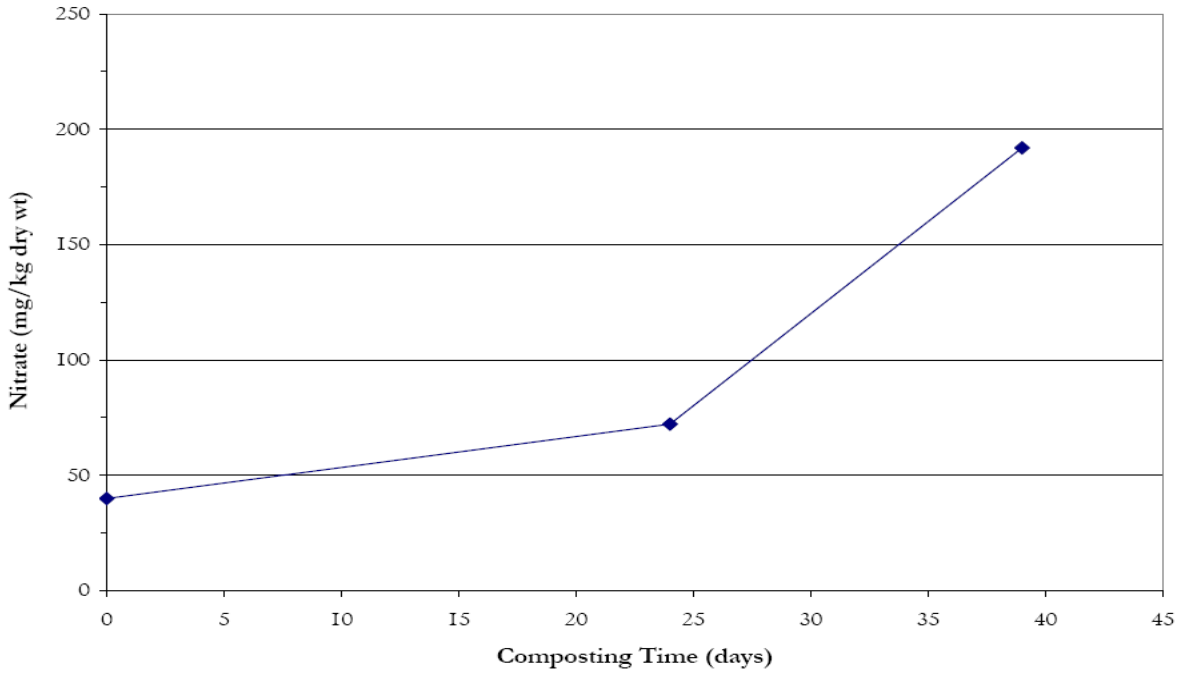


Figure 3.3-3: Nitrate-nitrogen concentrations during composting of 1M

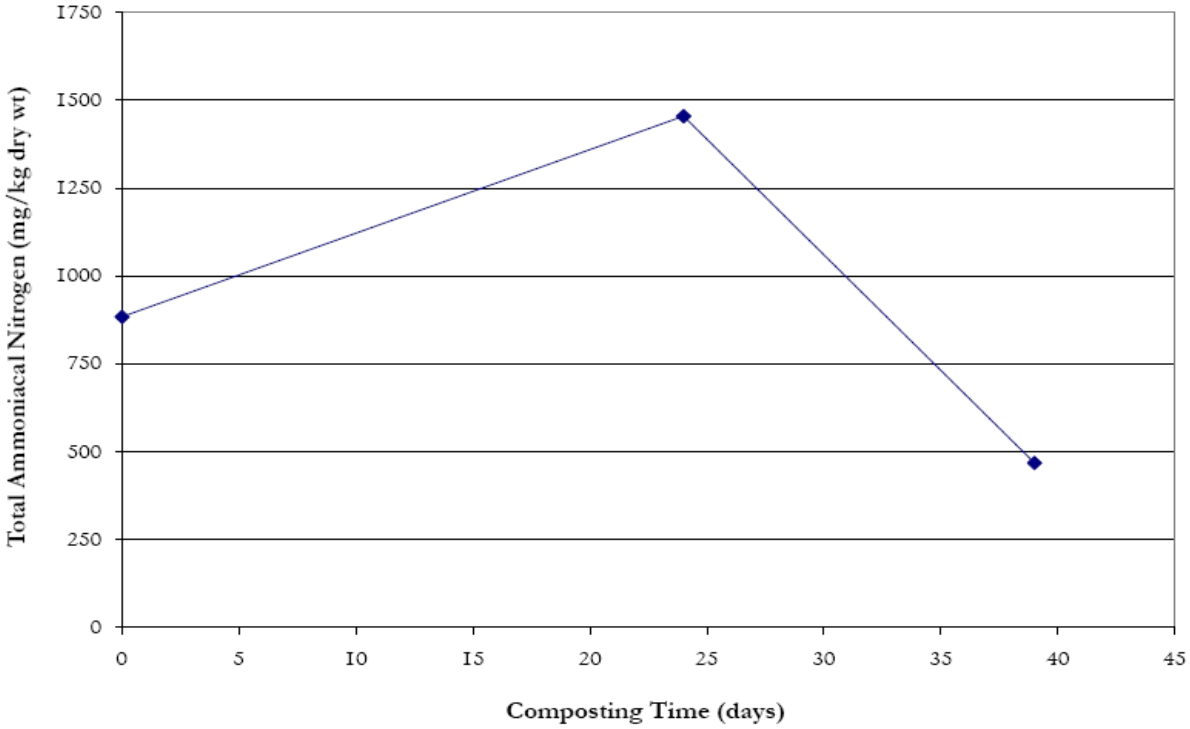


Figure 3.3-4: Total ammoniacal nitrogen concentrations during composting of 1M

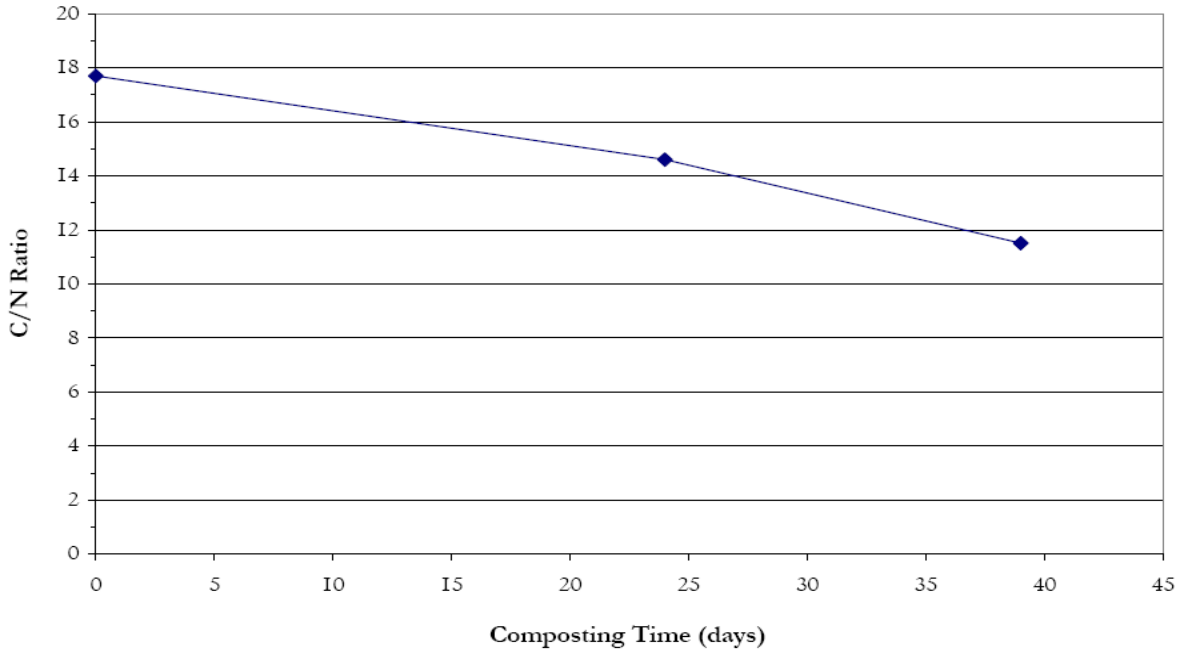


Figure 3.3-5: Carbon to nitrogen ratios for 1M during composting

Chapter 4
Materials and Methods

4.1 Composting

4.1.1 Compost Materials

Swine manure solids and *Paulownia* wood/leaves (bulking agent) were co-composted. The manure solids (White Oak Farms, Fremont, NC) were collected from an inclined screen separator as part of the evaluation of an advanced wastewater treatment system. Flushed waste from the confinement barns (fresh manure plus treated wastewater as flush water) flowed by gravity into a collection sump in which sat the lower end of the inclined screen. Rubber scrapper bars dragged suspended solids up the screen and into a manure spreader. The liquid that passed through the screen was pumped to an inclined plate clarifier in an attempt to remove additional fine solids. The liquid from this clarifier was then pumped to an aerobic treatment pond while the underflow was sent back to the collection sump and the inclined screen for removal. Effluent from the treatment pond was transferred to another inclined plate clarifier where biosolids were collected. The underflow from this clarifier was also pumped to the collection sump and the inclined screen separator for removal.

Flow into the collection sump consisted of 520 m^3/d with a suspended solids concentration of 5,080 mg/L from the confinement barns and 40 m^3/d with a suspended solids concentration of 17,000 mg/L from the biosolids clarifier. The suspended solids mass flow from the confinement barns was 2,640 kg/d and that from the biosolids clarifier was 680 kg/d. The cyclic flow between the collection sump and the manure clarifier was not measured because it was simply an internal cycle of the system. Characteristics of the manure solids (MN) are indicated in Table 4.2-4.

The solids were transported to the Animal and Poultry Waste Management Center (APWMC) at North Carolina State University (NCSU) for composting. The composting building was covered by a roof and contained a concrete floor; each compost pile was housed in a separate, partitioned "bin".

Paulownia trees (about 2 1/2 years old) were harvested at three different NCSU

experiment stations: Upper Piedmont Research Station, Reidsville; Oxford Tobacco Research Station, Oxford, and the Piedmont Research Station, Salisbury. Fallen leaves and tree branches, less than approximately 5 cm, were harvested. The harvested material was gathered in January and February and stored outdoors. The wood and leaves were chipped together (bulking agent), as needed.

The bulking agent was sized using sieving screens of 2.5 and 5 cm. On a weight basis, the bulking agent had the following size distribution: 1.7% > 5 cm, 2.5 cm < 1.9% < 5 cm, and 96.4% < 2.5 cm. However, the size distribution may be misleading since the chipping process often produced thin pieces with a high aspect ratio (length:width).

4.1.2 Composting Procedures

The windrow method was used as the composting procedure. This entailed piling the manure/bulking agent mix (feedstock) into rows and turning them periodically. Swine manure solids and chipped bulking agent were co-composted in a wet volume ratio of 1:1. The feedstock and bulking agent were mixed and later turned with a front-end loader, with supplemental manual mixing, as needed, to ensure well-mixed feedstocks. The initial size of the piles was approximately 2.5 m X 1 m X 0.65 m (length X width X height). The moisture content was maintained at a minimum of approximately 45%. This was achieved by a combination of periodic compost sampling and prior experience. Compost pile temperatures were measured at two depths: approximately 0.3 and 0.5 meters. For the first several weeks, the temperatures were monitored almost daily, then subsequently less frequently.

To evaluate different states of OM decomposition, four points of decomposition during composting were selected based on the typical temperature profile during composting, and Federal regulations, 40 CFR Part 503. In addition to the two end points of an uncomposted (initial mix) and a "fully composted" product (approximately 15 weeks), two other points within that range were chosen.

Normally, compost pile temperatures increase from ambient to thermophilic (> 45^{O} C) levels, remain there for a period of time, then decrease into the mesophilic (15-45^{O} C) range. The end of thermophilic decomposition is important for two reasons. First, thermophilic temperatures are necessary for inactivating many types of weed seeds and pathogenic

microorganisms, and second, decomposition rate is greatest during this stage. Preliminary work (Chapter 3) indicated that the duration of the thermophilic stage was approximately four weeks.

Additionally, the windrow composting process was governed by the objective of following EPA criteria for Class A "PFRP" with respect to pathogen reduction (Chapter 2.2.3.2). Consequently, the compost pile temperatures had to remain at least 55^{O} C for 15 days, with a minimum of 5 pile turnings within that time span. In addition to the two sampling points at the beginning and end of composting, the two additional time points selected were based on the compost temperature/time profile: 1) at the completion of the 15 day/55^{O} C PFRP requirement (designated SP) and 2) when the thermophilic phase was completed (designated MS). Instead of having specific sampling time points, these two supplemental data points were dependent upon composting conditions.

To obtain compost processed for the appropriate lengths of time, and so that the different end points coincided with a specific day for field application, the initiation dates for each compost treatment were staggered. The total time for the "fully composted" treatment (designated MT) was set at about 15 weeks, which was an estimate for producing a stable product, but also had practical significance for conducting the field trial during an appropriate growing season; the uncomposted feedstock (designated IN) was prepared on the day of field application The two intermediate composting points were initially estimated from composting MT with the assumption that the temperature profiles for all of the composts would be similar.

Three replicates per composting stage were produced, and each replicate was composted in a separate bin. The coding for the composts, including replicate designations (in parentheses) and their associated composting times was as follows:

SP (6, 13, 16) = 19 days
MS (7, 8, 9) = 39 days
MT (10, 11, 12) = 103 days
IN (1-3) = 0 days

4.1.3 Chemical Analyses

4.1.3.1 Sampling

Manure (MN) samples were collected on the day of field application. Samples of compost were collected during several points in the process. For the SP treatment, sampling was conducted on days 0, 5 and 19, which corresponded to the initial mix, the early stage of the thermophilic phase, and after the compost temperatures had maintained 55^{O} C for 15 days. For the MS treatment days 0, 4 and 39 were sampled, which corresponded to the initial mix, the early stage of the thermophilic phase, and the point when the compost temperatures had returned to mesophilic level. The MT treatment was sampled on days 0, 14, 49 and 103; these days were corresponded to the same processing points as MS, plus the final sampling, which represented stable compost. For all compost batches, the final sampling day, was the final composting day and the day of land application.

Approximately five grab samples were collected from each pile of manure or compost for each sampling day, and homogenized to obtain one composite sample per pile for analysis. Samples not analyzed within 24 hours were frozen.

4.1.3.2 Procedures

For pH and electrical conductivity measurements, samples were diluted 1:5 (weight: volume) with deionized water. The samples were allowed to sit for 15 minutes following dilution, then stirred, before taking measurements.

Moisture content was determined by heating samples at 105^{O} C overnight. Ash content was determined by heating the compost to 550^{O} C for 6 hours.

The Biological and Agricultural Engineering Environmental Analysis Laboratory (NCSU) performed the chemical analyses. Each sample, run in triplicate, was analyzed for total Kjeldahl nitrogen (TKN), nitrate-nitrogen, total ammoniacal nitrogen (TAN), available phosphorus, copper, zinc, total carbon and total nitrogen. TKN was determined using a persulfate digestion (Schuman et al. 1973) and analyzed per Standard Methods (1998) 4500Norg-B. Available phosphorus was extracted using a weak acid (Mehlich 1 extraction) (Olsen et al., 1982) and analyzed per Standard Methods (1998) 4500-P-F. TAN and nitrate (including nitrite) were extracted with K_2SO_4 (Keeney et al., 1982) and analyzed per Standard Methods (1998)

4500-NH3-G and 4500-NO3-E, respectively. For copper and zinc, compost samples were ashed at 500^{O} C, digested in concentrated acid, then analyzed using atomic absorption spectroscopy per Standard Method (1998) 3111-B; the manure samples were not ashed prior to acid digestion. Total carbon and nitrogen were determined by combustion, using a Leco C:N 2000 Autoanalyzer (Leco, St. Joseph, MO). All of the samples were run in the "as-is" state.

4.1.3.3 Organic Acid Analysis

4.1.3.3.1 Phenolic Acids

The extraction procedure and sample preparation for spectrophotometric analysis were based on the techniques described by Blum et al. (1991).

I. Extraction

The aqueous extraction procedure was as follows:

1) the compost sample (10-20 g) was diluted with deionized/distilled water in the ratio of 11 water :1 compost on a dry weight basis
2) autoclaved for 45 minutes, in glass bottles with tight fitting caps, on liquid cycle
3) the bottle was mechanically shaken for approximately 30 minutes at room temperature, to enhance cooling
4) the liquid was poured off and centrifuged in Teflon® tubes for 10 minutes at relative centrifugal force, $RCF_{MAX} = g \text{ X } 26916$
5) the supernatant was filtered through a 20 μm pore filter (Whatman #41), then a nominal 0.45 um pore, glass fiber filter (Gelman Metrigard), using a vacuum pump

II. Absorption

A spectrophotometric technique (Spectronic 401 spectrophotometer; Spectronic Instruments, Inc., Rochester, NY) was used to measure total phenolic substances (Swain et al., 1959). The procedure (Blum et al., 1991) entailed mixing 5 ml of standard or 0.5 ml of compost extract plus 4.5 ml of sterilized (autoclaved) deionized water with 0.75 ml of 1.9 M Na_2CO_3 and 0.25 ml of Folin and Ciocalteu's phenol reagent. The mixture was incubated for 2 hours in the dark at room temperature. Absorption was measured at 725 nm (Marambe et al., 1992). (A

preliminary investigation indicated no differences in absorption when the measurements were taken at 725 nm or 750 nm.) Total phenolic content was measured based on a 4-hydroxycinnamic acid standard (Garcia et. al, 1991a; Marambe et. al, 1992). The compost extracts were either measured as-is or diluted 1:1 with sterilized deionized water. Recovery rates were measured for 4-hydroxycinnamic acid and phenol.

4.1.3.3.2 Fatty Acids

Microbac Laboratories, Inc., Southern Testing and Research Division, Wilson, NC, performed the analysis for the saturated fatty acids, C2 – C18, plus C18:3. (The coding for fatty acids was as follows: C4, for example, indicates that the saturated fatty acid is a 4 carbon chain, and C18:3 means 3 double bonds occur in the aliphatic chain.) Excluding acetic acid (C2), the fatty acid contents were determined by capillary gas chromatography (AOAC 996.06). The basic steps were acid extraction of the samples, ether extraction of the acid extract, derivitization of the fatty acids by esterification, then analysis using gas chromatography. Acetic acid concentrations were determined by titration (AOAC 942.15). All samples were run in duplicate.

The samples analyzed represent the initial and endpoints of each of the treatments:

MN (manure): day 0
IN treatment: day 0
SP treatment (6, 13, 16): days 0 and 19
MS treatment: (7, 8, 9): days 0 and 39
MT treatment (10, 11, 12): days 0 and 103

4.1.4 Calorimetry

An adiabatic (constant temperature) bomb calorimeter (Model 5500, IKA, Wilmington, NC) was used to determine the calorific values of the manure and compost. Frozen samples were defrosted in a refrigerator, dried at 70^{O} C overnight, then ground to $\leq$ 0.5mm with a Wiley mill. Using a pellet press, pellets were made for the calorimeter using about 1 g of the ground samples. Certified benzoic acid pellets (IKA, Wilmington, NC) were used to calibrate the calorimeter, which was performed each day of analysis.

Gross calorific values were calculated based on ASTM method D5468-02, including

making adjustments for nitric and sulfuric acid formation. For the latter, the sulfur content of the samples was needed. Sulfur content was determined on the ground samples following the procedure by Novozamsky et al. (1986) and was performed by the Soil Analytical Laboratory, Department of Soil Science.

Nitric acid formation was determined by titration based on ASTM D240-02. After sample combustion, the calorimeter bomb apparatus was rinsed with 60 ml of deionized water. Five drops of 0.1% bromophenol blue solution was added to this water, which was then titrated with a standardized 0.1N NaOH solution (Standard Methods, 1995). Two sodium hydroxide solutions were made during the calorimetric analysis, and neither solution was used for longer than 14 days. Preliminary analysis had indicated that a 0.1N NaOH solution was stable for at least 18 days.

Calorific values were calculated based on dry weight and ash free content. Moisture content was determined by drying the ground samples at 105^{O} C overnight. The ash contents of the ground samples were determined by heating the samples to 550^{O} C for six hours.

The following equation was used to calculate gross calorific values:

$$((10675 * T_R) - e2 - N - S)/W = J/g \text{ sample} \qquad (4\text{-}1)$$

10675 = average calorific values of benzoic acid samples, $J/^{o}$ K
T_R = temperature rise of bomb due to sample combustion, ^{o} K
e2 = energy from electric ignition and consumable ignition wire, J
N = correction factor for nitric acid level, J
S = correction for sulfur concentration, J
W = sample weight, g

The value 10675 was the average of the daily calibrations that were performed. Nitrogen and sulfur correction factors were necessary due to the exothermic reactions of the formation of NO_x and SO_2/SO_3 during combustion and the subsequent formation of nitric and sulfuric acids due to hydrolysis with water (heats of solution). The formation of HNO_3 and H_2SO_4 release higher amounts of energy than just the oxidation of nitrogen and sulfur, respectively. Therefore, correction factors are used to adjust the calculations for this extra heat production.

4.2 Field Study

4.2.1 Design

The field study was designed for a one season investigation, and was conducted at the Lake Wheeler Road Field Laboratory (Appendix A). Grain sorghum was selected (Mueller, personal communication) primarily because of its drought tolerance. When initially planning the field study, it was uncertain whether irrigation would be available for the study that was to begin in June.

The investigation involved seven soil treatments: 4 compost amendments (IN, SP, MS, MT), a swine manure amendment (MN), and unamended soil (NF) and fertilized soil (F) controls. Each soil treatment was a separate plot, with three replicates per treatment, for a total of 21 plots. The plot size (4 X 4 meters) was primarily dictated by the availability of compost.

For statistical analysis purposes, the plots were arranged in a randomized complete block design, with the rows being the blocking factor. The blocking factor was to account for the slope in the landscape, the rows being perpendicular to the slope. The field design consisted of three rows of seven plots with 3 meter buffers between rows, and a 1 meter buffer between individual plots (Figure 4.2-1). Each plot contained four rows of sorghum, with a 0.5 meter unsown perimeter.

The soil was an Appling series fine, sandy loam classified as a thermic, typic, kaolinitic, Ultisol, Kanhapludult. The application rates of the soil amendments and fertilizers were based on a soil analysis (North Carolina Department of Agriculture and Consumer Services - NCDA & CS - Raleigh, NC) (Table 4.2-1). For the fertilizer treatment (F), nitrogen was applied at 101 kg/ha and potash at 67 kg/ha per recommendation; other nutrients were at adequate levels. For growing sorghum, recommendations for nitrogen application ranged from 140-220 lbs/ac depending upon its end usage; for potash, 110-130 lbs/ac were recommended (NCDA & CS, personal communication). However, these recommendations assume three cuttings of sorghum per year. For one cutting, as in the present study, application rates of 90 lb N/ac (101 kg/ha) and 60 lb K_2O/ac (67 kg/ha) were suggested (NCDA & CS; Randy Weisz, Department of Crop Science, NCSU).

Lower Elevation

IN3		MN1		F3		MS7		MT11		SP6		NF2
MT12		NF3		MS9		F2		IN1		MN3		SP13
SP16		MS8		IN2		MT10		MN2		F1		NF1

Higher Elevation

Figure 4.2-1: Field layout. The soil treatments were composts IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day), manure (MN), fertilizer (F) and unamended (NF) control. The numbers following the soil treatment designation indicate replicate coding: IN (1-3), SP (6, 13, 16), MS (7-9), MT (10-12), F (1-3) and NF (1-3).

Table 4.2-1: Selected Soil Characteristics

Characteristics	**Value ***
Density	1.28 g/cm^3
CEC	4.2 meq/100 cm^3
Base Saturation	81 (% CEC)
Acidity	0.8 meq/100 cm^3
pH	5.8
Phosphorus	117 mg/kg (dry wt)
Potassium	58 mg/kg (dry wt)
Calcium	61 (% CEC)
Magnesium	15 (% CEC)
Copper	6 mg/kg (dry wt)
Zinc	6.6 mg/kg (dry wt)
Sodium	0

* based on Appendix B and assuming 15 cm plow depth

Although the fertilized controls received the recommended nutrients, the additions of manure and compost were based on the total nitrogen levels in the amendments that would be become bioavailable during the growing season, via nitrogen mineralization. Since the actual nitrogen mineralization rates were unknown prior to the field study, a literature review (Appendix C) provided guidance in hypothesizing mineralization rates based on initial amounts of organic nitrogen in the amendments. Additionally, the amendment application rates were not based on day-of-application chemical analyses, which were not practical, but on analyses close to the application date or other compost batches (Table 4.2-2). Therefore, the nutrient loading rates of the amendments were based on the amendment application rates (Table 4.2-3) and the organic amendment characteristics (Table 4.2-4). Nutrient loading rates are indicated in Table 4.2-5.

Calcitic lime was applied to the field site, at a rate of 2 t/a, to increase the soil pH to about 6 (Larry King, personal communication). The lime was applied approximately 2 months prior to sowing. The whole field was disked several days prior to planting.

On the day of planting, the soil amendments were applied to the whole 4 m X 4 m plots, then disked into the soil to a depth of about 15 cm. Following soil preparation, the sorghum seed was sown at a rate of about 1 seed/5 cm length, with 1 m row spacing. Four rows per plot were sown. No herbicides or pesticides were applied before or during the experiment. Weeding was performed manually. The sorghum was irrigated with 2.5 cm of water if no measurable rain had accumulated within the previous week.

Table 4.2-2: Total nitrogen content of organic amendments: based on day-of-application analyses (Nitrogen Content II) and previous analyses (Nitrogen Content I) on which amendment application rates were based

Soil Amendment	Nitrogen Content I (previous analysis) (% dry wt)	Nitrogen Content II (day-of-application analysis) (% dry wt)
MN manure	2.3 a	1.8
IN compost	2.0 b	1.6
SP compost – 6	2.2 c	2.7

Table 4.2-2 (continued)		
SP compost – 13	2.2 c	2.5
SP compost – 16	2.2 c	2.3
MS compost – 7	3.0 d	3.3
MS compost – 8	3.0 d	3.5
MS compost – 9	3.0 d	3.5
MT compost – 10	3.3 e	3.3
MT compost – 11	3.2 e	3.2
MT compost – 12	3.3 e	3.3

a: average of manures from 4/27/99 and 5/18/99
b: average based on initial mixtures of SP and MS composts
c: based on analyses of MT composts after 2 weeks
d: based on MT compost after pile temperatures had returned to mesophilic stage
e: analyses performed 1 week prior to application

Table 4.2-3: Application rates of soil amendments: composts IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day), manure (MN), fertilizer (F) and unamended (NF) control. The numbers following the soil treatment designation indicate replicate coding: IN (1-3), SP (6, 13, 16), MS (7-9), MT (10-12), F (1-3) and NF (1-3).

Soil Amendment	**Plot (Replicate) Numbers**	**Nitrogen Content (% dry wt)**	**Predicted Mineralization Rate (%)**	**Application Rate (kg/plot)***	**Application Rate (Mg/ha)***
MN	1-3	2.3	30	24	15
IN	1-3	2.0	25	33	21
SP	6	2.2	20	37	23
SP	13	2.2	20	37	23
SP	16	2.2	20	37	23
MS	7	3.0	15	36	22.5
MS	8	3.0	15	36	22.5
MS	9	3.0	15	36	22.5

Table 4.2-3 (continued)					
MT	10	3.3	8	62	39
MT	11	3.2	8	64	40
MT	12	3.3	8	62	39
F	1-3	-	-	**	**

* dry weight basis applied to 4 m X 4 m plot area

** TAN-nitrate fertilizer and potash applied at 0.16 kg/plot (101 kg/ha) and 0.11 kg/plot (67 kg/ha), respectively

Table 4.2-4: Average nutrient concentrations of organic amendments on day of field application

	Organic Amendment				
Characteristic (dry wt)	**MN**	**IN**	**SP**	**MS**	**MT**
TN (%)	1.8	1.6	2.5	3.4	3.3
TKN (mg/kg)	16512	15422	22493	33625	30145
NH_4-N (mg/kg)	1775	450	504	149	236
NO_3-N (mg/kg)	1.3	0.3	33	107	2451
OM (%)	85	82	80	78	68
Phosphorus (mg/kg)	5889	18403	12864	12968	26874
Copper (mg/kg)	431	409	367	414	670
Zinc (mg/kg)	2186	1421	1795	2284	3370
Moisture Content (%)	71	65	46	44	52

Table 4.2-5: Nutrient Loading Rates (application rate X amendment concentration on day of application) on a per hectare basis for organic amendments: manure (MN) and composts (IN, SP, MS and MT)

	Organic Amendment				
Characteristic	**MN**	**IN**	**SP**	**MS**	**MT**
dry wt	**kg/ha**				
TN	270	336	575	765	1287
TKN	248	324	517	757	1176
NH_4-N	27	9	12	3	9
NO_3-N	0.02	0.006	0.8	2	96
OM (Mg/ha)	13	17	18	18	27
Phosphorus	88	386	296	292	1048
Copper	6	9	8	9	26
Zinc	33	30	41	51	131

4.2.2 Harvest

All the plots were not harvested on one date. Each plot was harvested as the sorghum grain began drying and reached a specific moisture content, the benchmark moisture content being determined at harvest. However, the objectives were to obtain mature grain and also minimize grain consumption by birds, which inferred an early harvest.

Grain was obtained from the periphery of the plot (outside of the "harvest" area) to determine grain moisture prior to harvesting the plants. Harvesting consisted of cutting the sorghum stalks at soil level. From the middle two rows, 2.75 m of each row from the center of the plots were collected. Several sorghum plants, at each end of the 2.75 m harvested portion, were also collected for determining the moisture content of the grain (dried overnight at 105^{O} C). Following harvest the field was allowed to remain fallow.

Immediately following harvest, the plants were dried at approximately 60^{O} C for 36 hours to minimize decomposition. Subsequently, the grain was manually separated from the stalk and sieved through a series of screens (4.5, 3, 1.5 mm) to remove extraneous material. The dry matter remaining on the sieving screens was added to the above ground harvested plants to obtain total stover biomass.

Total nitrogen of the grain and stover were determined with a Leco C:N 2000 Autoanalyzer (Leco, St. Joseph, MO). Prior to analysis, the sorghum was dried at a stable 70^{O} C for 24 hours due to inconsistent temperatures in the initial drying oven, and ground to less than 1 mm using a Wiley Mill. Nitrogen concentrations were normalized to 12% moisture content; stover yields were based on dry weights.

4.3 Soil Properties

4.3.1 Sampling

Samples were obtained from the three intra-crop rows, to a depth of 15 cm, the approximate plow depth. Samples were taken from plot locations that were undisturbed by previous sampling. Soil samples were collected for up to a full year: June 1999 to June 2000, although the duration and frequency were not same for each analysis. The samples collected at harvest were collected the day following crop harvest of each plot.

4.3.2 Chemical Analyses

Samples for pH and electrical conductivity measurement were collected on weeks 1, 3, 6, 10, harvest, 28, 39 and 52. For electrical conductivity measurements, the samples were diluted 1:5 (dry weight:volume), with deionized water. Samples were diluted 1:1 (dry weight: volume) with deionized water, for pH measurements. After dilution, samples were allowed to sit for about 15 minutes, and stirred once during that time period, prior to analysis.

The BAE Environmental Analysis Laboratory performed the nutrient analysis. The nutrients of interest were TKN, nitrate, available phosphorus, calcium and magnesium. TKN, nitrate, available phosphorus were analyzed as described in Section 4.1.3. Calcium and magnesium were extracted using a weak acid (Mehlich 1) and detected using atomic absorption spectroscopy (NCDA & CS, Agronomic Division, Soil Testing Methods, prior to July, 1981).

4.3.3 Physical Properties

4.3.3.1 Bulk Density

Samples were obtained four times over a 12 month period: 1 week, 10 weeks, harvest (~ 3 months) and 12 months. Coring cylinders of 10.2 cm diameter were used to obtain the initial (June) measurements. Thereafter, cylinders of 7.6 cm diameter were used. Three samples per plot were obtained. The samples were weighed, dried at 105^{O} C overnight, then reweighed. The soil was sieved to remove stones greater than 1.25 cm. Calculations for bulk density accounted for the stones, assuming a stone density of 2.65 g/cc.

4.3.3.2 Penetration Resistance

Soil penetration resistance was measured using a Soiltest Proving Ring penetrometer (ELE International LLC, Soiltest Products Division, Loveland, CO). Four measurements were taken on each plot. Measurements were taken 5 days, and 11, 12 and 17 weeks following amendment application.

4.3.3.3 Water Holding Capacity

The water holding capacity at -0.01 MPa (field capacity) and –1.5 (wilting point) MPa were determined for the soil after 2 weeks, 10 weeks and harvest. The procedures were

performed based on Klute (1986), and the water content of the samples was determined by drying them at 70^{O} C for 24 hours. For field capacity measurements, intact soil cores were obtained, and refrigerated until use. For wilting point determinations, soil samples were air dried, then sieved to obtain aggregates less than 2 mm.

4.3.3.4 Aggregate Stability

Soil samples were collected 5 days, 2 months, harvest, and 6, 9 and 12 months following amendment application. Two samples per plot were collected with a coring device, and homogenized, then air dried, and sieved to obtain aggregates 1-2 mm. The procedures for wet sieving were similar to those described by Yoder (1936); Haynes et al. (1997) also used a procedure similar to the one used in the present study. A multiple sieving apparatus was used with screen sizes of 1 mm, 500 μm, 250 μm and 105 μm. Six sets of sieves were operated simultaneously.

Aggregate stability determination was a 2-step process. The first step consisted of wet sieving the aggregates; the second step involved disintegrating the aggregates and separating out the primary soil particles and large pieces of organic matter. The procedure was as follows:

1. about 20-25 grams of air dried aggregates were placed on the 1 mm screen
2. the aggregates were wetted by lowering the screen into the water bath (25-28^{O} C) such that the water was at the height of the screen; the aggregates were allowed to absorb water for 5 minutes, for a fairly rapid wetting; this technique was similar to Elliot (1986)
3. aggregates were wet sieved for 30 minutes, with an oscillation rate of 30 cycles/min, and amplitude of approximately 5 cm
4. the water was drained from the bath; the sieves were drained for several minutes
5. the sieves, plus aggregates were oven dried at 105^{O} C overnight, then reweighed
6. using gentle force and forced water, the aggregates were dispersed on each sieve
7. the sieves, plus soil, were oven dried, as before, then reweighed

4.4 Nitrogen Mineralization Study

4.4.1 Procedures

An incubation study was conducted to determine the nitrogen mineralization potential of five soil amendments: These were the same treatments used in the field study: manure (MN) and four composts (IN, SP, MS, MT). Unamended soil was used as the negative control.

Soil from the field study site was collected and mixed with the amendments. Sample cores, of 15 cm depth, were taken and homogenized then sieved to obtain aggregates less than 2 cm. Two hundred grams of soil (dry weight) were mixed with the appropriate amount of amendment. Since composts SP, MS and MT, were produced in triplicate, equal dry weights from each replicate compost pile were mixed together to obtain a homogenized amendment mix for each treatment, prior to mixing with the soil.

The amendments were mixed with soil at a higher application rate than in the field study because of inhomogeneity of the composts. The relatively large pieces of chipped bulking agent (wood/leaves) compared to the manure created inhomogeneous mixtures in the early stages of decomposition (ie, composting). The inhomogeneity was magnified, compared to the field study, due to the relatively small amount of soil (200 g) used in this incubation study. The amendments were mixed with soil at an equivalent rate of 3360 kg N/ha (1750 mg/kg dry wt) (Appendix D).

The soil and amendments were mixed on a dry weight basis and placed in plastic bags that were subsequently sealed. The mixtures were incubated in the dark at 23^{O} C for up to 16 weeks, which approximated the growing season for sorghum. The moisture content of the bags was checked weekly, by weighing the bags and comparing the results to the original weights. Deionized water was replenished as needed. During the weighing procedure, all of the bags were opened briefly.

The initial moisture contents of the mixtures were calculated to achieve a total water quantity equivalent to approximately 50% moisture content in the amendment and approximately 95% container capacity in the soil (Appendix D). Container capacity was determined by saturating the soil with deionized water, allowing the soil to drain for 24 hours with the weights determined before and after drying.

With amendments SP (46% moisture) and MT (52% moisture) containing approximately 50% moisture, water was added to achieve 95% container capacity. For MS (44% moisture), in

addition to adding container capacity water, water was also added to attain 50% moisture level in the amendment. Although the moisture content of IN was 65%, when the total water content of the amendment/soil system was normalized to 95% soil container capacity, IN contained approximately 51% moisture, which is consistent with moisture contents of SP, MS and MT. MN contained 71% moisture. Normalizing the amendment/soil system to 95% soil container capacity, resulted in an amendment moisture content of 60% (Appendix D).

Samples were analyzed each week in triplicate, for total carbon, total nitrogen, TAN, nitrate-nitrogen and moisture content per Section 4.1.3.2. For moisture content, samples were dried at 105^{O} C overnight.

4.4.2 Calculations

To assess the production of inorganic nitrogen (nitrate, nitrite and TAN) total inorganic nitrogen, net inorganic nitrogen and percent mineralized nitrogen were calculated. Total inorganic nitrogen (eq. 4-2) was the summation of nitrate, nitrite and TAN, and deemed plant available nitrogen. Net inorganic nitrogen (eq. 4-3) was the calculated difference between the inorganic nitrogen concentrations in the amendment soil/mix and the control soil. Percent mineralized nitrogen (eq. 4-4) was the amount of inorganic nitrogen produced by the organic amendment based on the extant levels of organic nitrogen at the beginning of the experiment. The mathematical expressions for calculating total inorganic nitrogen (INT), net inorganic nitrogen (NIN) and percent mineralized nitrogen (PMN) were as follows:

$$INT = (NO3_T + NO2_T + TAN_T) \quad (4\text{-}2)$$

$$NIN = INT_{MT} - INT_{ST} \quad (4\text{-}3)$$

$$PMN = \{[(INT_{MT} - INT_{M0}) - (INT_{ST} - INT_{S0})]/(TN_{M0} - INT_{M0})\} \text{ X } 100 \quad (4\text{-}4)$$

NO3 = nitrate nitrogen
NO2 = nitrite nitrogen
(legend continues next page)

TAN = total ammoniacal nitrogen
TN = total nitrogen
M = compost/soil mix
S = soil
T = time T
0 = time 0

4.5 Statistical Analyses

SAS (SAS, Cary, NC) software was used for statistical analyses. For the results of the chemical and thermodynamic characterization of the composts (Chapters 5.1.3 – 5.1.6), a random effects (analysis of variance) model, with treatment and time as the fixed effects and treatment replicate the random effect (Proc Mixed, SAS) was used to analyze the data. Statistical analyses were used to compare amendments, measure the effect of time and make comparisons between the initial and final measurements for each treatment, and between the final values for every treatment. One degree of freedom contrasts were used to make specific pairwise comparisons. In some cases (described in Chapter 5), the data was mathematically transformed to make the statistical model residuals more homogeneous (Kleinbaum et al., 1988).

For the nitrogen mineralization study results, a random effects model (analysis of variance), with treatment and time as the fixed effects and sample replicate as the random effect (Proc Mixed, SAS) was used. For net inorganic nitrogen and percent mineralized nitrogen, treatment comparisons at 16 weeks were made using Tukey's (HSD) multicomparison test. For C:N ratios and total nitrogen concentrations, one degree of freedom contrasts were used for comparing the values at 0 and 16 weeks.

Except for soil water holding capacity, repeated measures of analysis (Proc GLM, SAS) was the statistical procedure used for analyzing the data sets for soil nutrients and soil physical properties as sampling occurred over a period of time from the same plots. The variables treatment and row were the main effects. For each data set, if the conditions for sphericity were not met (Mauchly W test), the Huynh-Feldt correction was used to adjust the probabilities (Kuehl, 1994).

For water holding capacity, a split plot statistical model was used instead of repeated measures of analysis because of missing data points. Split plot models are able to accommodate

missing measurements, whereas repeated measures of analysis models are not. The variables treatment, time and row were the main effects.

For the plant response data, the analysis of variance model (Proc GLM, SAS) included treatment and row (blocking factor) as fixed effects. Differences between treatments were determined using Tukey-Kramer multicomparison procedure.

Three distinct probability notations will be used throughout discussions of statistical analyses: Pr means probability, Pr_{MAX} indicates maximum probability, and Pr_{MIN} indicates minimum probability. Summaries of the statistical analyses can be found in Appendix H.

Chapter 5
Results and Discussion

5.1 Compost Characterization

5.1.1 Overview

In the ensuing discussion concerning compost characteristics, one must keep in mind that the initial compositions of the four composts (IN, SP, MS and MT) were not identical. While the swine manure and bulking agent were consistently mixed in a 1:1 volume ratio, the composition of the bulking agent changed. With each successive compost batch (MT→MS→SP→IN), the ratio of leaves:wood chips decreased (visual observation). Additionally, since each compost batch was processed at a different time of the year, the climatic conditions (eg, ambient temperature and precipitation) differed and may have affected the composting dynamics. Therefore, differences between the four composts may have been related to the slightly different proportions of the same feedstock (Michel et al., 1996; Gunnarsson et al., 2002; Cambardella et al., 2003; Huang et al., 2004) and the composting conditions (Parkinson et al., 2004; Tiquia, 2005).

Numerous compost characteristics have been proposed as indices for determining the appropriate duration of composting (Chapter 2.3). In the present study, many of the compost characteristics that were determined were also potential stability indices. Therefore, the discussions of compost characteristics will also be viewed within the context of these indices. While stability refers to the potential for high microbial activity, the terms stability and maturity (ie, degree of humification) have been used interchangeably, too often. Therefore, although the ensuing discussion involves stability indices, these parameters may also include those originally designated for "maturity".

5.1.2 Compost Pile Temperatures

Figure 5.1-1 shows the average treatment temperature profiles for the three composts: SP, MS and MT. The last two average temperature measurements for MT at 86 days (21.2^{O} C) and 103 days (20.7^{O} C), the day of application, were not graphed for enhanced clarity.

Additionally, for simplicity, the data was graphed to show the process initiation of the three compost batches occurring at about the same time. However, in actuality, to obtain compost processed for the appropriate lengths of time, and so that the different end points coincided with a specific day for field application, the initiation dates for each compost treatment were staggered.

While the temperatures were originally measured at both 30 cm and 46 cm depths, only the 30 cm depth was reported. Initially, the two depths approximated 1/2 and 2/3 respectively, of the height of the compost piles. However, as decomposition progressed, the compost piles compressed, and eventually the 46 cm depth was the total pile height in some instances. Thus, the 30 cm depth was the most representative of the pile temperature.

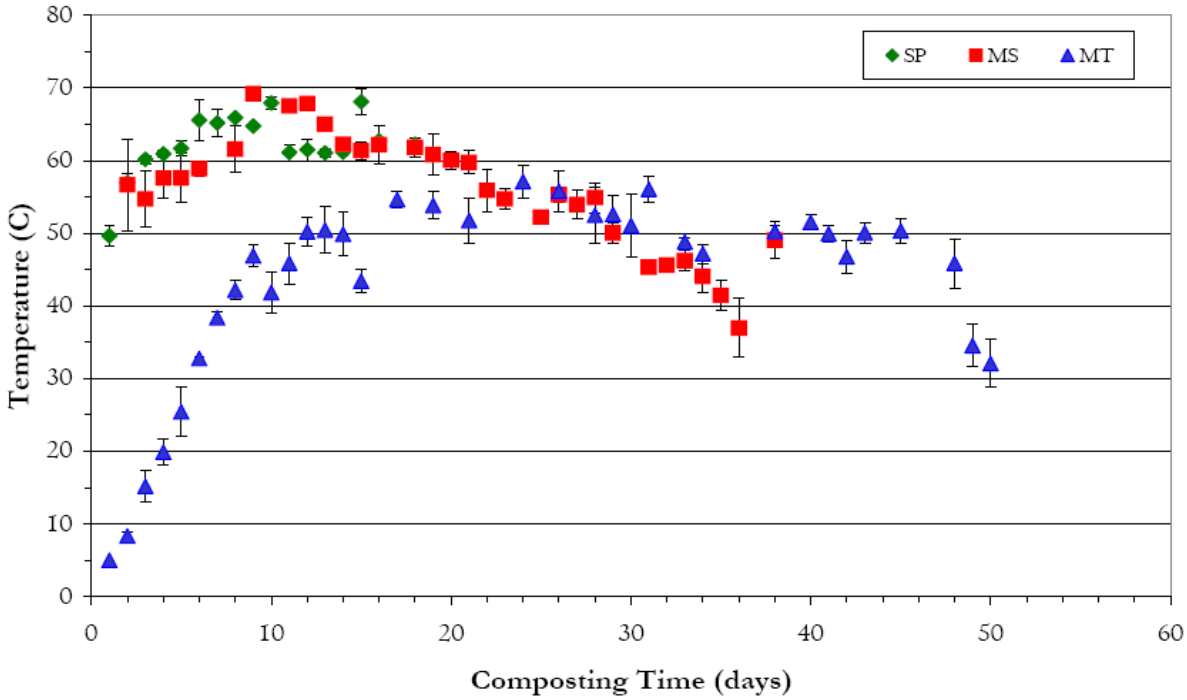


Figure 5.1-1: Average compost pile temperatures from a 30 cm depth for SP (◆), MS (■) and MT (▲). Error bars are 1 standard deviation.

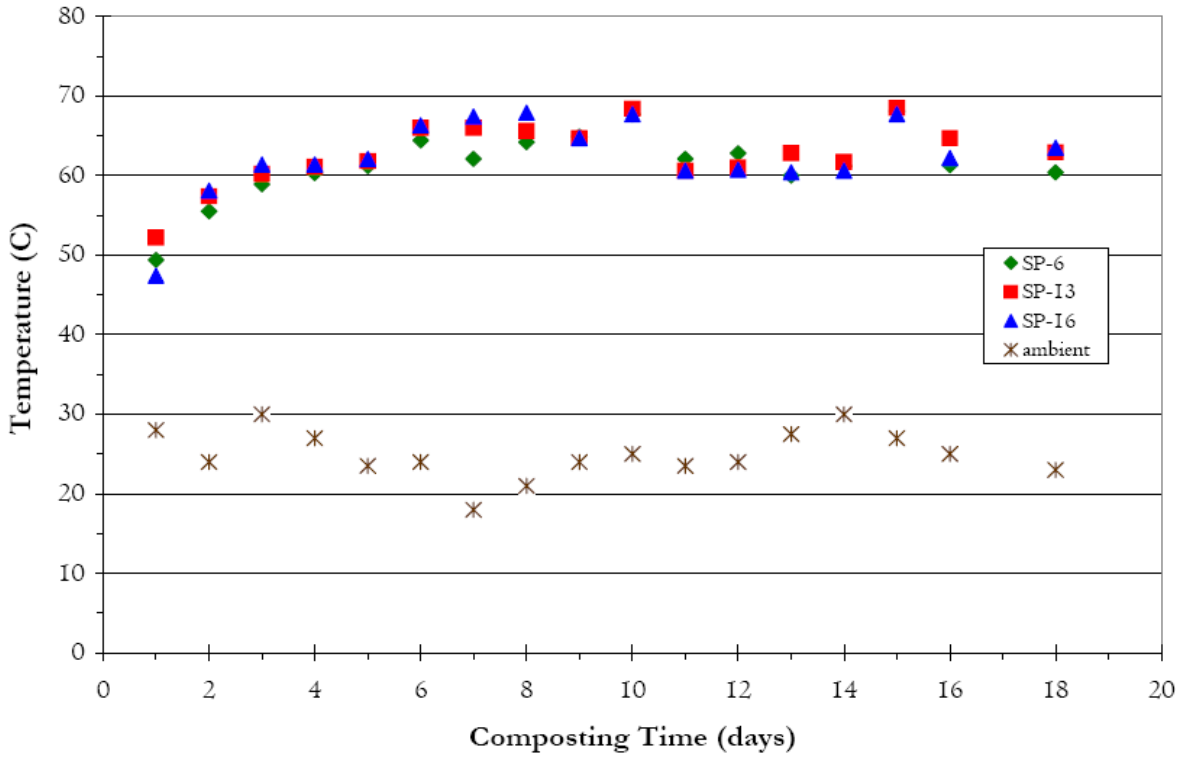


Figure 5.1-2: Temperature profiles for replicate piles 6 (◆), 13 (■) and 16 (▲) of compost SP and ambient temperature (✳). Compost temperature measurements were taken at a 30 cm depth. To conform to Federal regulations CFR 40 Part 503, the piles were turned 5 times during the period when the temperatures were at least 55^{O} C. The turning days were 5, 7, 9, 14 and 16.

Achieving a Class A product was successful for SP and MS, but only partially so for MT. For SP (Figure 5.1-2) and MS (Figure 5.1-3), the temperatures for each replicate pile of each compost batch remained above 55^{O} C for at least 15 days (during which time all the piles were turned at least 5 times). However, for MT, only replicate pile 12 was deemed Class A, as the temperatures for the other two replicate piles did not remain above 55^{O} C for at least 15 days (Figure 5.1-4).

The reason that two of the three MT replicates did not attain sufficiently high temperatures (and the third replicate did not exceed the minimum PFRP requirements) appears to have been related to the low ambient temperatures. During the period of composting MT, especially the early stages, the ambient temperatures were lower than the ambient temperatures

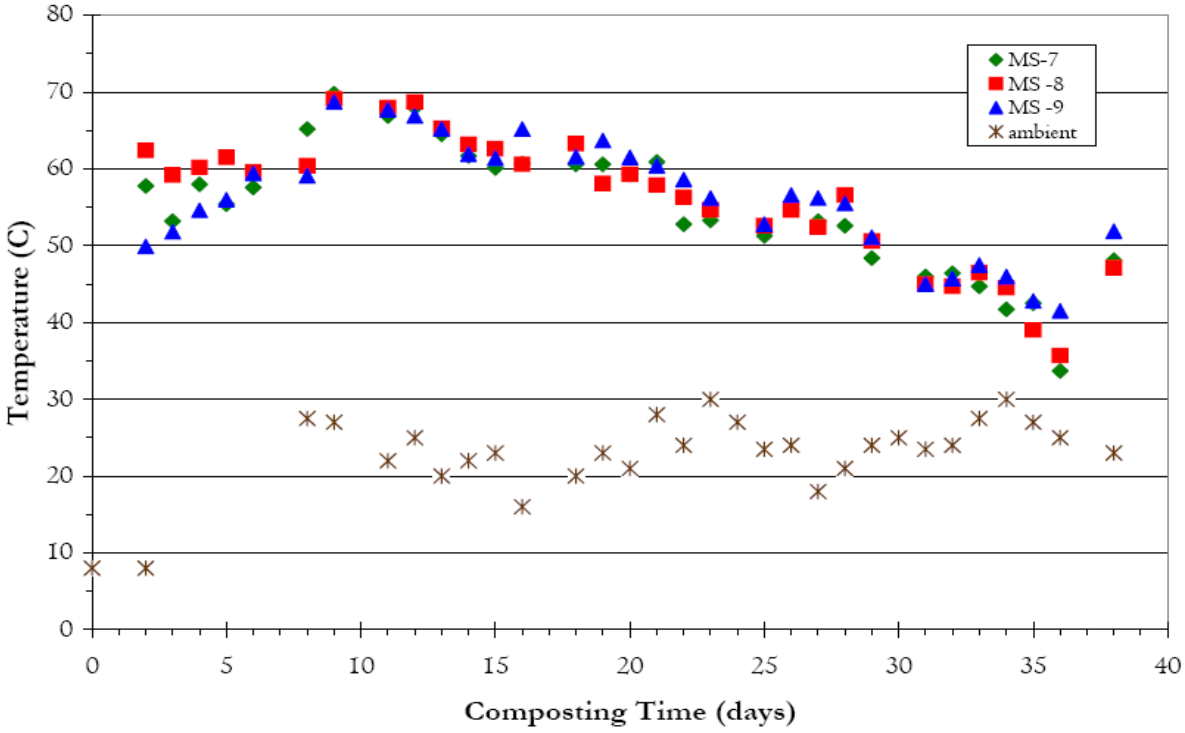


Figure 5.1-3: Temperature profiles for replicate piles 7 (◆), 8 (■) and 9 (▲) of compost MS and ambient temperature (✳). Compost temperature measurements were taken at a 30 cm depth. To conform to Federal regulations CFR 40 Part 503, the piles were turned at least 5 times during the period when the temperatures were at least 55^{O} C. The turning days were 4, 6, 8, 11, 15 and 18.

during composting of SP and MS. The lower temperatures may have depressed microbial activity, and also cooled the MT piles to a greater extent than when composting SP and MS.

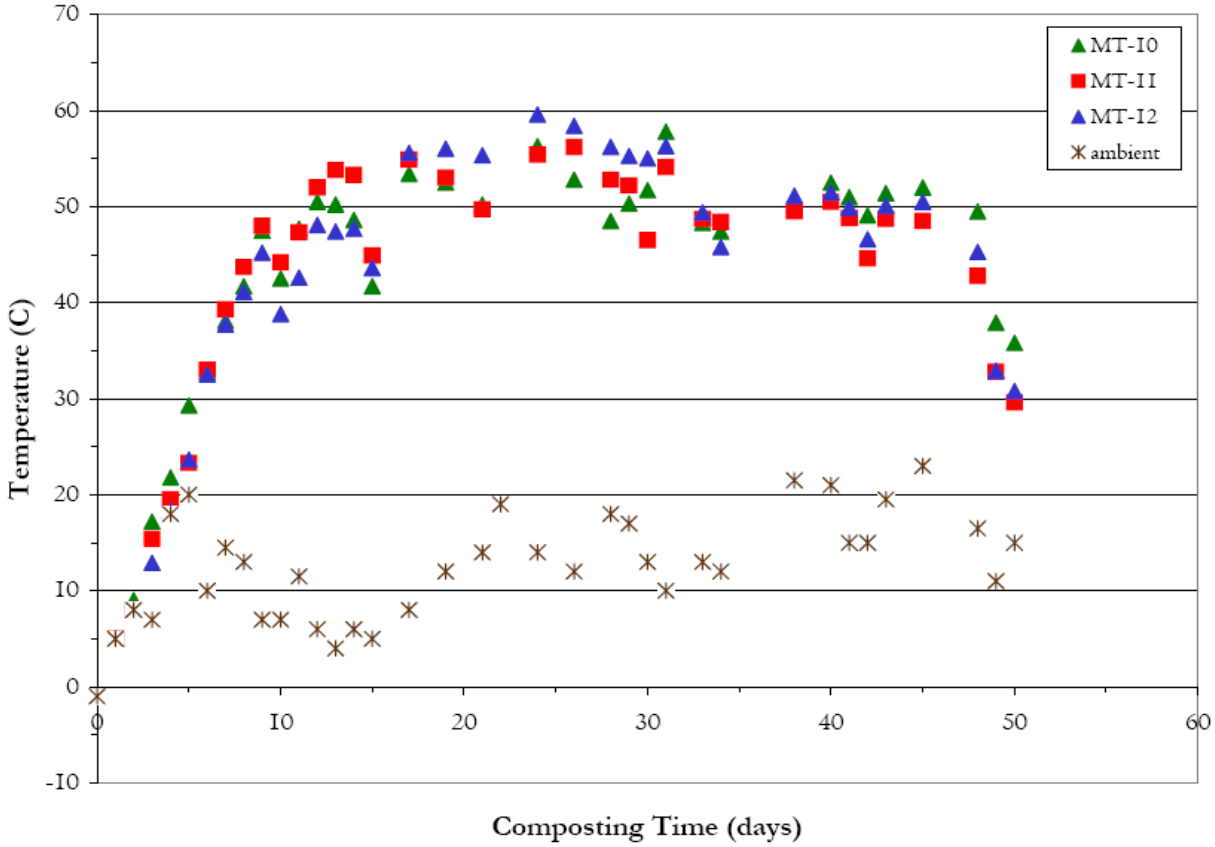


Figure 5.1-4: Temperature profiles for replicate piles 10 (◆), 11 (■) and 12 (▲) of compost MT and ambient temperature (✳). Compost temperature measurements were taken at 30 cm depth. To conform to Federal regulations CFR 40 Part 503, the piles were turned at least 5 times during the period when the temperatures were at least 55^{O} C. The turning days were 17, 22, 26, 29 and 31.

5.1.3 Compost Nutrients

5.1.3.1. C:N Ratio

Figure 5.1-5 shows the C:N (total carbon/total nitrogen) ratios of all of the soil amendments. The C:N ratios of each compost batch decreased with composting duration, and were reflections of the combined dynamics of the carbon and nitrogen transformations.

Statistically, the main effects, time and treatment, were statistically significant (Pr_{MAX} = 0.0001) For SP, MS and MT, the initial and final ratios for each treatment, were significantly different (Pr_{MAX} = 0.0001). Comparing the final treatment ratios, all of the ratios were significantly different (Pr_{MAX} = 0.003).

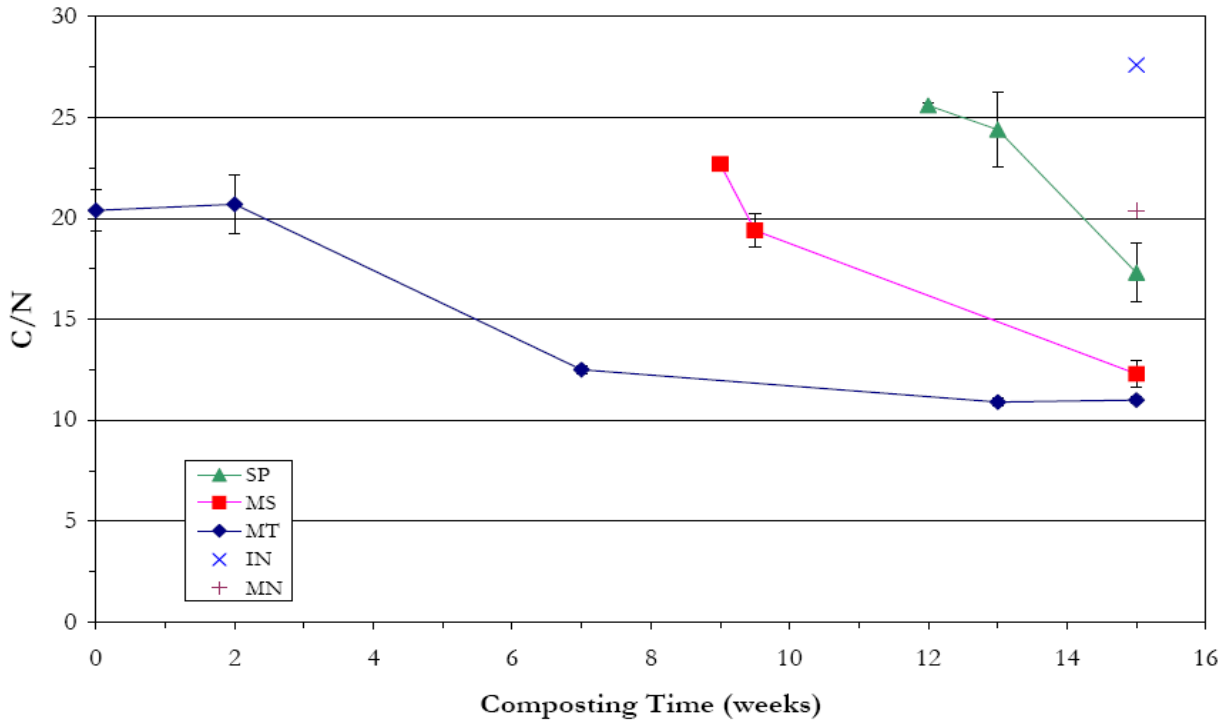


Figure 5.1-5: C:N ratios for treatments SP (▲), MS (■), MT (◆), IN (×) and MN **(+).** Error bars are 1 standard deviation.

With each successive compost batch (MT➔MS➔SP➔IN), the initial C:N increased, which was primarily an indication of the changing composition of the bulking agent, paralleling the increasing proportion of wood, which has a higher C:N ratio than leaves (Poincelot, 1975). Consequently, the addition of the bulking agent to the manure increased the C:N ratios for all compost batches. For MT, the C:N ratio increased from about 17.4 (manure) to 20.4 (mix), from 20.5 (manure) to 22.7 (mix) for MS, from 20.0 (manure) to 25.6 (mix) for SP, and from 20.4 (manure, MN) to 27.5 (mix, IN). Additionally, the C:N ratios of the manure varied during the year. Values of C:N of samples taken monthly, January to June excluding February, ranged from 17.4 to 20.5, with a C:N of 20.5 for the manure that was applied to the field.

The C:N ratio is a commonly used indicator of composting progress, and has been numerically indexed to denote stability. This will be discussed further in Chapter 5.1.6. However, one weakness of using the C:N ratio as an assessment of compost stability is when using

feedstocks of initially low C:N. A C:N*t* /C:N*i* ratio (t indicating time t, and i denoting initial for C:N ratios) was suggested as a guide for measuring composting progress (Morel et al., 1979), as well as an alternative to the composting index of final C:N (Morel et al., 1987), although no numerical values were indexed. There is poor correlation between this ratio and composting time on a universal scale and therefore (C:N)*f*/(C:N)*i* would not be an absolute indicator of stability, although this ratio may be applicable as a complementary stability index (Iglesias-Jiménez et al., 1989). Figure 5.1-6 indicates that for compost MT in the present study, there was an inverse relationship between this ratio and time, and that the values leveled off as composting time increased. Therefore, this ratio may be an appropriate complementary index for these feedstocks.

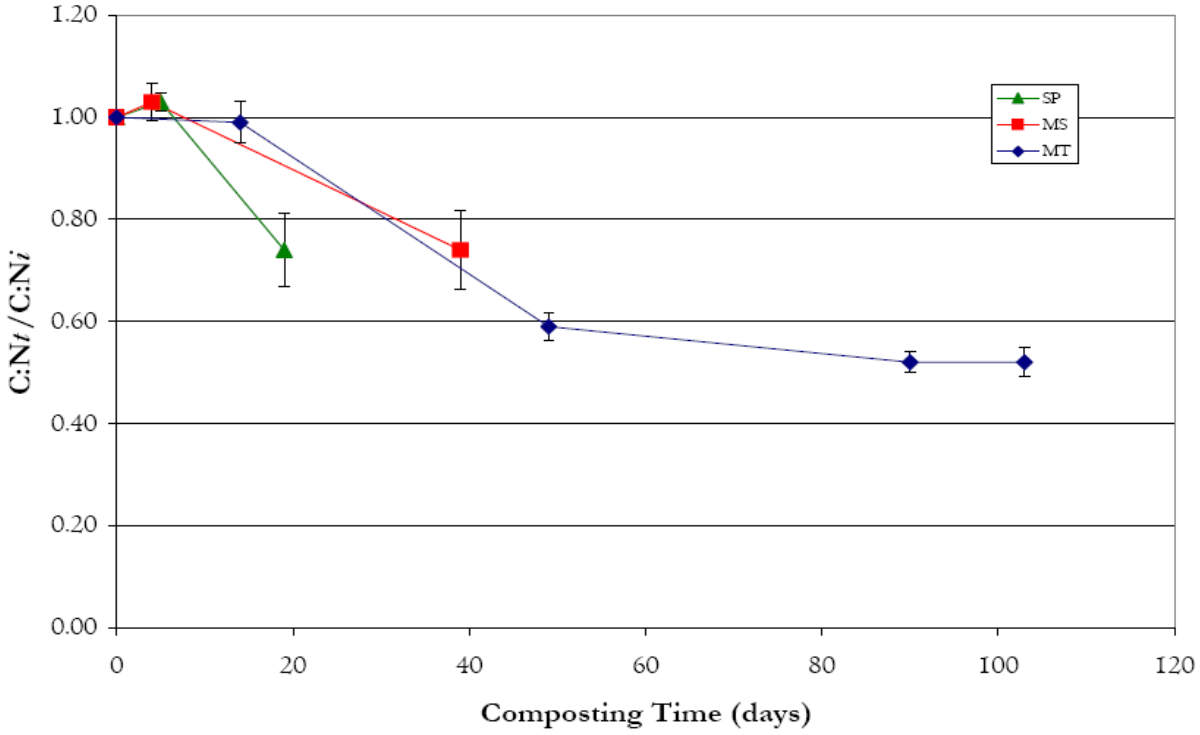


Figure 5.1-6: C:N*t*/C:N*i* ratios of composts SP (▲), MS (■) and MT (◆). Error bars are 1 standard deviation.

5.1.3.2 Nitrogenous species

Figure 5.1-7 shows the progression of total Kjeldahl nitrogen (TKN) based on either dry weight or ash free, dry weight. The oscillating trends that occurred were the combined effects of losses of inorganic nitrogen via gaseous emission (Witter et al., 1988; Martins et al., 1992 Eghball et al., 1997), leachate runoff (Ott et al., 1983; Martins et al., 1992; Nienaber et al., 1994; Eghball et al., 1997) and possibly fixation of atmospheric nitrogen (Diaz-Raviña et al., 1989); juxtaposed on nitrogen transformations, was carbon dioxide losses due to carbon mineralization, increasing the apparent nitrogen concentrations. The similarity of the SP and MS curves may reflect the similar temperature profiles, and thus decomposition dynamics, which were dissimilar to MT.

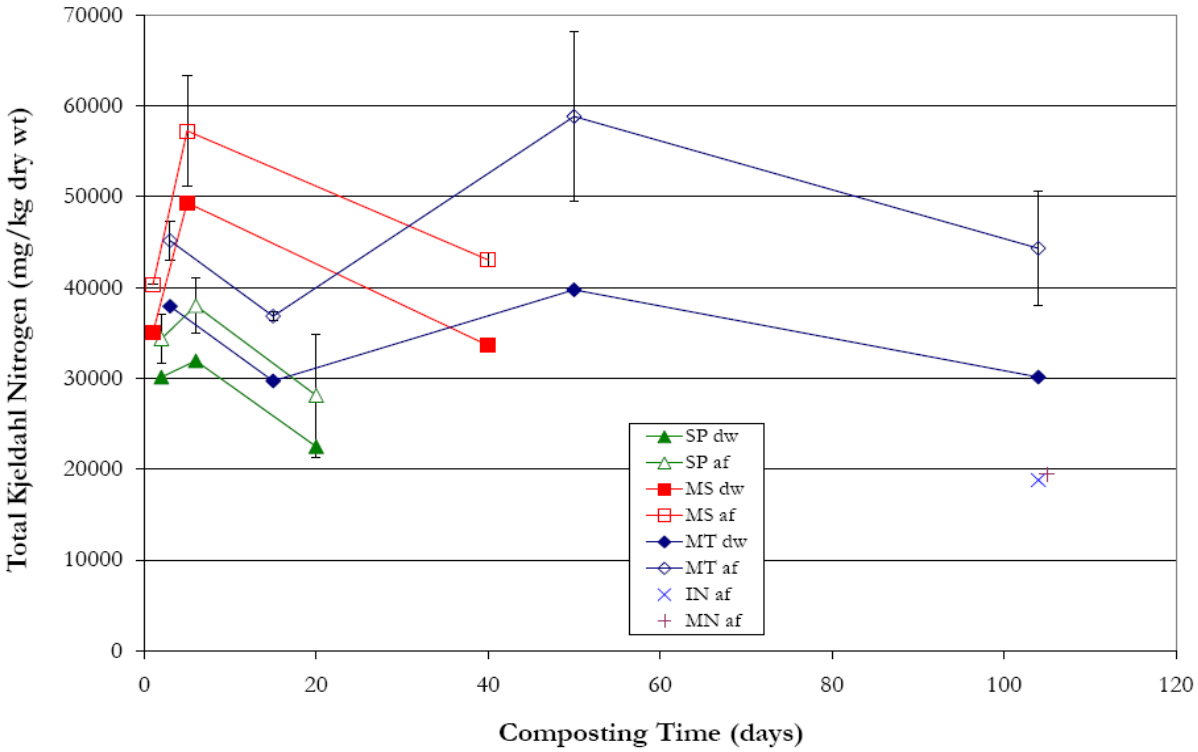


Figure 5.1-7: Total Kjeldahl nitrogen for treatments SP (▲), MS (■), MT (◆), IN (×) and MN (+). Solid symbols are on dry weight bases (dw). Open symbols, plus IN (×) and MN (+) are on ash free dry weight basis (af). Initial time for SP, MS and MT are offset for clarity. Error bars, which are associated with the open symbols, are 1 standard deviation.

The data for total Kjeldahl nitrogen was square root transformed to obtain more homogeneous residuals for statistical analysis. However, the data presented in Figure 5.1-7 are the untransformed values. The statistical analysis was based on the ash free, dry weight data. The main effects, treatment and time, were statistically significant (Pr_{MAX} = 0.001). The initial and final TKN levels for each compost, SP, MS and MT, were statistically equivalent (Pr_{MIN} = 0.087). Comparing final concentrations, the following treatments were statistically equivalent (Pr_{MIN} = 0.737): MN-SP and MS-MT. All other pairwise comparisons between final concentrations were statistically different (Pr_{MAX} = 0.009): The total Kjeldahl nitrogen was comprised primarily of organic nitrogen since the inorganic nitrogen levels (Figures 5.1-8 and 5.1-9) were at least an order of magnitude less than TKN.

The data for total ammoniacal nitrogen concentrations were log (LN) transformed to obtain more homogeneous residuals for statistical analysis, which was based on the ash free, dry weights. However, the data presented in Figure 5.1-8 are the untransformed values. The overall effects of treatment and time were statistically significant (Pr_{MAX} = 0.0001). The initial and final total ammoniacal nitrogen concentrations for SP were statistically equivalent (Pr = 0.407); for each treatment, MS and MT, the initial and final concentrations were statistically different (Pr_{MAX} = 0.0001). For pairwise comparisons of the final concentrations, IN-MT and IN-SP were statistically equivalent (Pr_{MIN} = 0.055); all other pairwise comparisons were statistically different (Pr_{MAX} = 0.014).

The data for nitrate concentrations was log (LN) transformed to obtain more homogeneous residuals for statistical analysis, which was based on the ash free, dry weights. However, the data presented in Figure 5.1-9 are the untransformed values. The main effects, treatment and time, were statistically significant (Pr_{MAX} = 0.0001). Comparing the initial and final nitrate concentrations for treatments SP, MS and MT indicated that only in SP were the initial and final levels statistically equivalent (Pr = 0.052); for the other treatments, the initial and final concentrations were significantly different (Pr_{MAX} = 0.0001). For pairwise comparisons of the final concentrations, only IN-SP was statistically equivalent (Pr = 0.669); all other pairwise comparisons were statistically different (Pr_{MAX} = 0.0001).

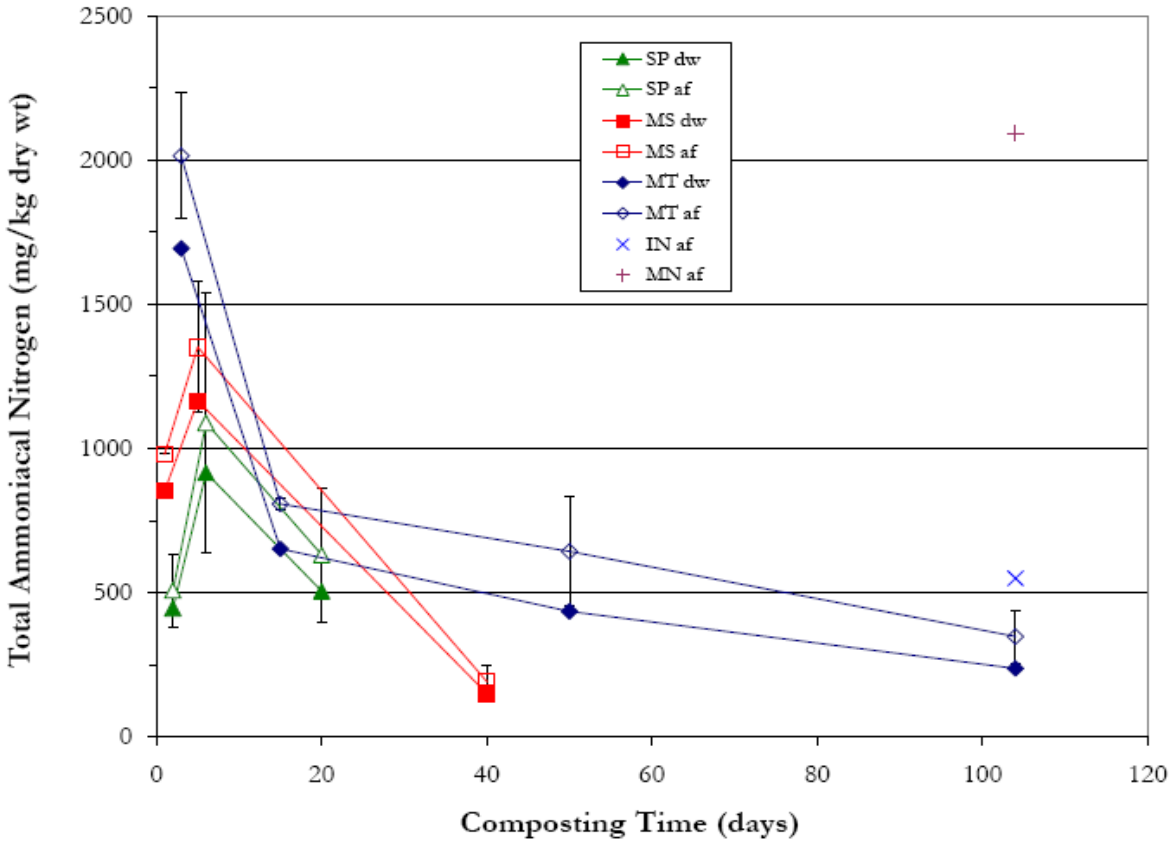


Figure 5.1-8: Total ammoniacal nitrogen for treatments SP (▲), MS (■), MT (◆), IN (×) and MN (+). Solid symbols are on dry weight bases (dw). Open symbols, plus IN (×) and MN (+) are on ash free dry weight basis (af). Initial time for SP, MS and MT are offset for clarity. Error bars, which are associated with the open symbols, are 1 standard deviation.

The decreases in TAN and increases in nitrate, shown in Figures 5.1-8 and 5.1-9, respectively, are common trends in composting (Riffaldi et al., 1986; Mahimairaja et al., 1994; Tiquia et al., 1998; Huang et al., 2004). As ammonia volatilization is often the primary loss mechanism for nitrogen during composting (Martins et al., 1992; Eghball et al., 1997), ammonia volatilization would have been enhanced by the alkaline pH of the composts (Figure 5.1-19), since volatilization is promoted by alkaline conditions ($NH_3 + H_2O \leftrightarrow NH_4^+ + OH^-$). However, the temperature profiles of the compost piles are also indicative of other factors affecting TAN concentrations. The similarity of the TAN temporal profiles for SP and MS, which were both dissimilar to MT, may have been due to the different temperature profiles. The

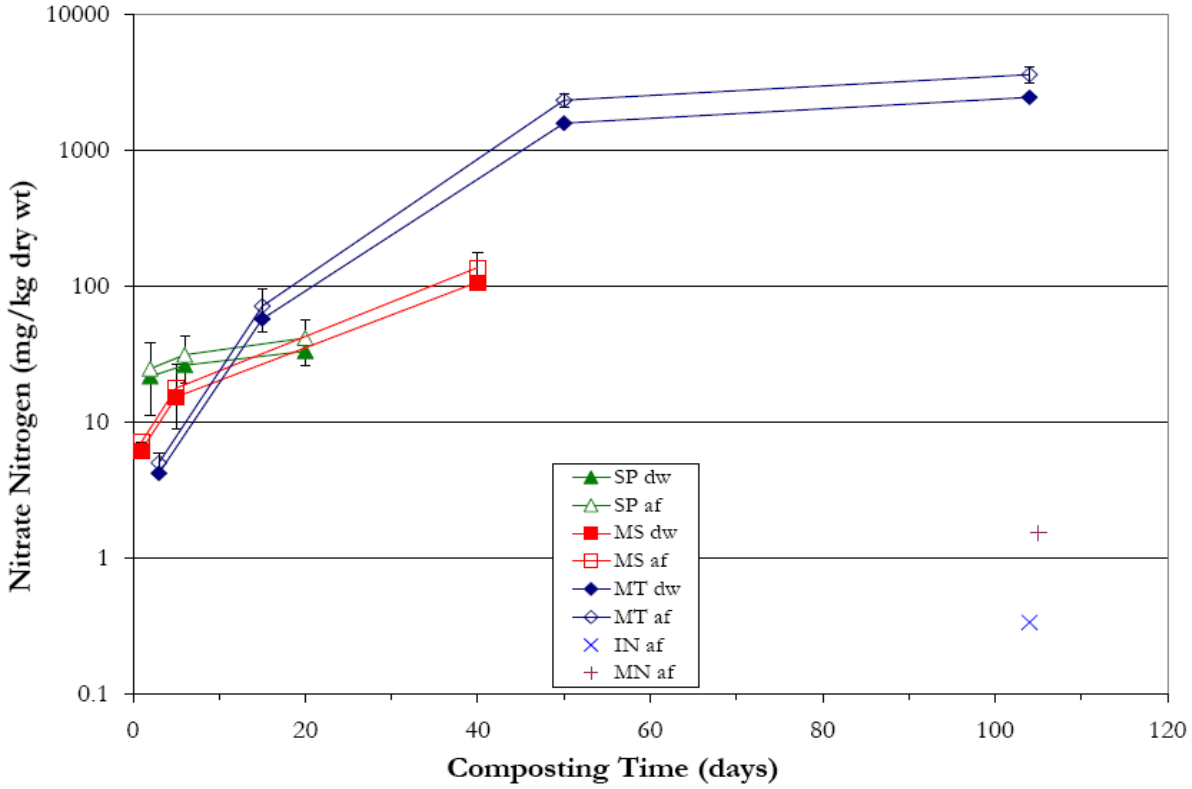


Figure 5.1-9: Nitrate-nitrogen for treatments SP (▲), MS (■), MT (◆), IN (×) and MN (+). Solid symbols are on dry weight bases (dw). Open symbols, plus IN (×) and MN (+) are on ash free dry weight basis (af). Initial time for SP, MS and MT are offset for clarity. Error bars, which are associated with the open symbols, are 1 standard deviation.

temperature profiles for SP and MS indicated a sharp rise in temperature, which would have enhanced ammonia volatilization. However, the corresponding increase in TAN levels was similar to the increase in TKN and therefore may have only been an apparent increase due to substrate loss, although increased nitrogen ammonification due to increased microbial activity may also have been a contributing factor. The subsequent declines may have been due to increased ammonia volatilization. Ammonia is insoluble in water above 60^{O} C and although the compost was not chemically analyzed as frequently as the compost temperatures were monitored, maximum TAN concentrations for SP and MS occurred at about the same time that the compost temperatures attained about 60^{O} C (Figures 5.1-1, 5.1-2 and 5.1-3), followed by a decline. Decreased microbial activity, which begins to decline at about 60^{O} C (Chang et al., 1967;

Ladd et al., 1972), may have been a factor in the sharp decline in total ammoniacal rapid nitrogen concentrations; depletion of nutrients, reducing microbial activity, may have contributed to the longer term decline of TAN levels. For MT (Figures 5.1-1 and 5.1-4), the slower temperature rise resulted in different decomposition dynamics, with an initial sharp decline of TAN followed by a more moderate decrease. Although the frequency of compost sampling was low, the demarcation between the rapid and moderate decrease generally corresponded to the time when the pile temperature attained thermophilic temperatures (> 45^{O} C). The initial rapid decline of TAN (and corresponding increase in nitrate) may have been partially related to the temperature threshold for nitrification.

Nitrifying microbes are inhibited above 40^{O} C (30 – 35^{O} C is optimum) (Paul et al., 1996), and this may have been the reason that MT had a greater rate of nitrate increase (and conversely TAN decrease) than SP or MS (Figure 5.1-9). MT took a longer time to achieve thermophilic temperatures, and achieved maximum temperatures much lower than SP and MS, whereas SP and MS temperatures sharply increased and remained significantly above mesophilic (45^{O} C) for extended periods. For MT, the curve indicates three different nitrate accumulation rates, which approximated three temperature phases of the composting process (Figure 5.1-1 and 5.1-4): rise to thermophilic temperature, the thermophilic stage and the mesophilic phase again. An additional discussion of nitrogen dynamics can be found in Chapter 5.2.

Concentrations of TAN and nitrate have been suggested as composting indices, and numerical values have been recommended as indicators of stability (Chapter 5.1.6). As composting progresses, TAN concentrations tend to decrease and nitrate levels increase. Consequently, the TAN/nitrate ratio tends to decrease (Figure 5.1-10). The decreasing rate of change as composting progresses indicates that this ratio may be an appropriate composting index. Numerical values for this index will be discussed in Chapter 5.1.6.

5.1.3.3 Organic Matter

Organic matter is the nutrient source for microbes. As decomposition progresses, nutrients are consumed and are transformed into more recalcitrant forms, with the consequence that microbial activity declines. During composting, organic matter losses are high in the early stages of decomposition, then tend to decrease as readily available nutrient sources become

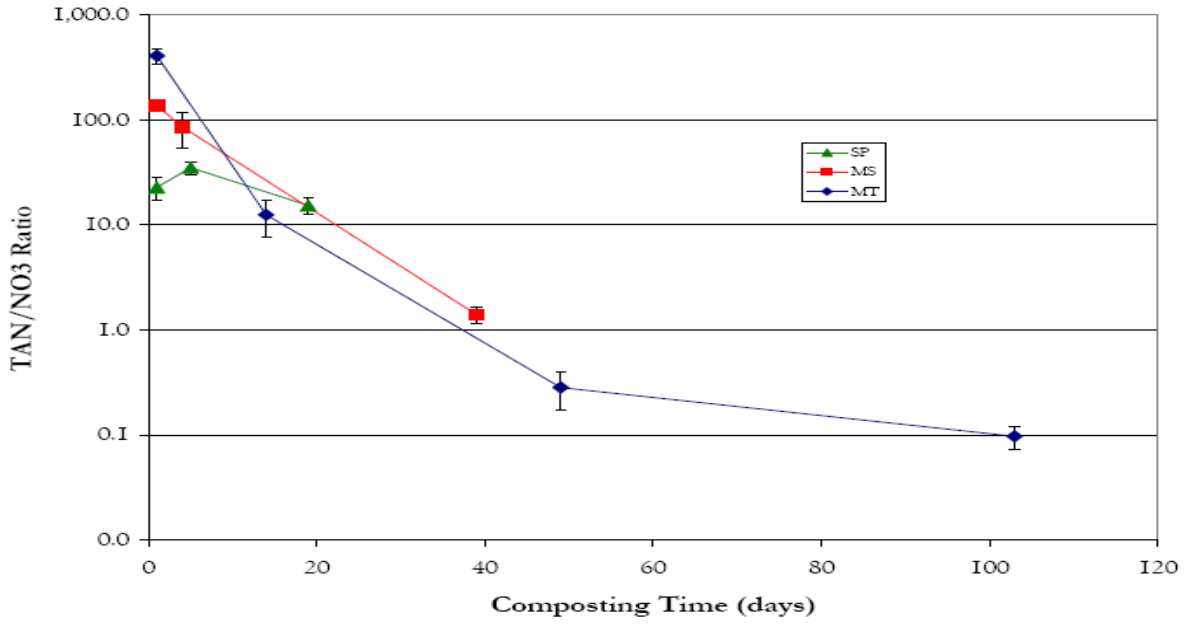


Figure 5.1-10: Ratio of total ammoniacal nitrogen (TAN) to nitrate (NO3) for composts SP (▲), MS (■) and MT **(◆).** Error bars are 1 standard deviation.

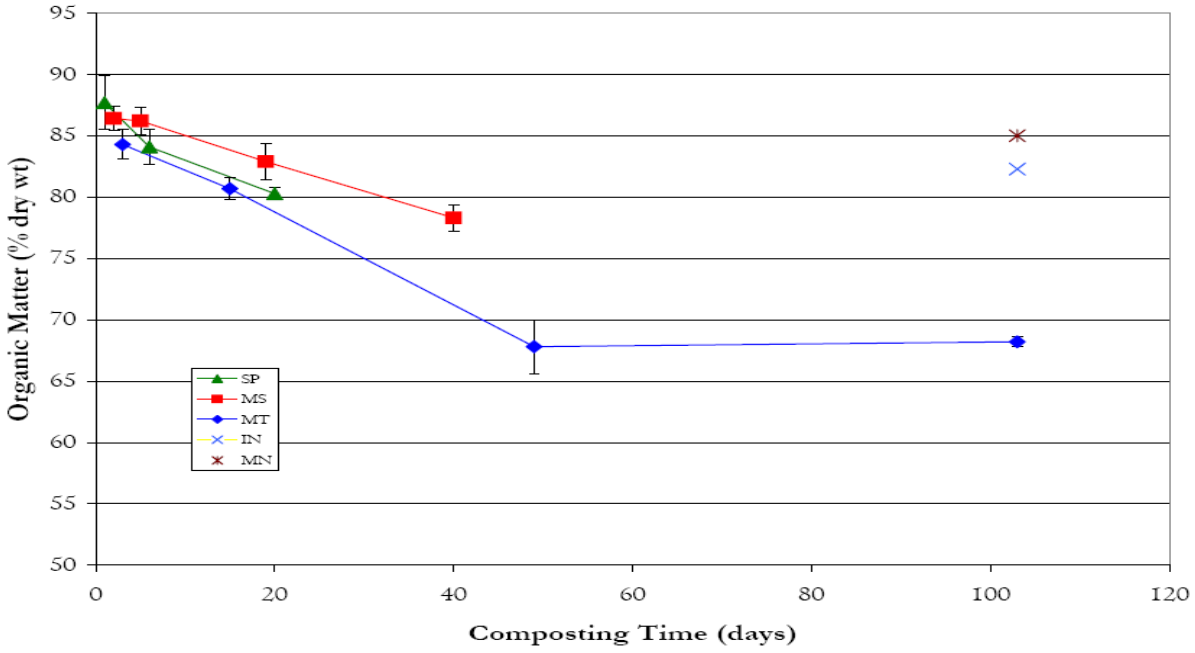


Figure 5.1-11: Organic matter content in organic amendments SP (▲), MS (■), MT (◆), IN (×) and MN (✳). Initial sampling times may be offset for clarity. Error bars are 1 standard deviation.

depleted (Figures 5.1-11). Low, stable OM content has been suggested as a complementary index for composting stability (Ouatmane et al., 2000).

5.1.3.4 Phosphorus

The data set for Mehlich 1 extractable phosphorus concentrations was log transformed to obtain more homogeneous residuals for statistical analysis. However, the data presented in Figure 5.1-12 are the untransformed values. The variables treatment and time had overall statistically significant effects (Pr_{MAX} = 0.0001). The initial and final concentrations for each compost (SP, MS and MT) were statistically different (Pr_{MAX} = 0.0005). Comparing all treatments, the final concentrations were statistically different (Pr_{MAX} = 0.0002) except for SP-MS, which were statistically equivalent (Pr = 0.830).

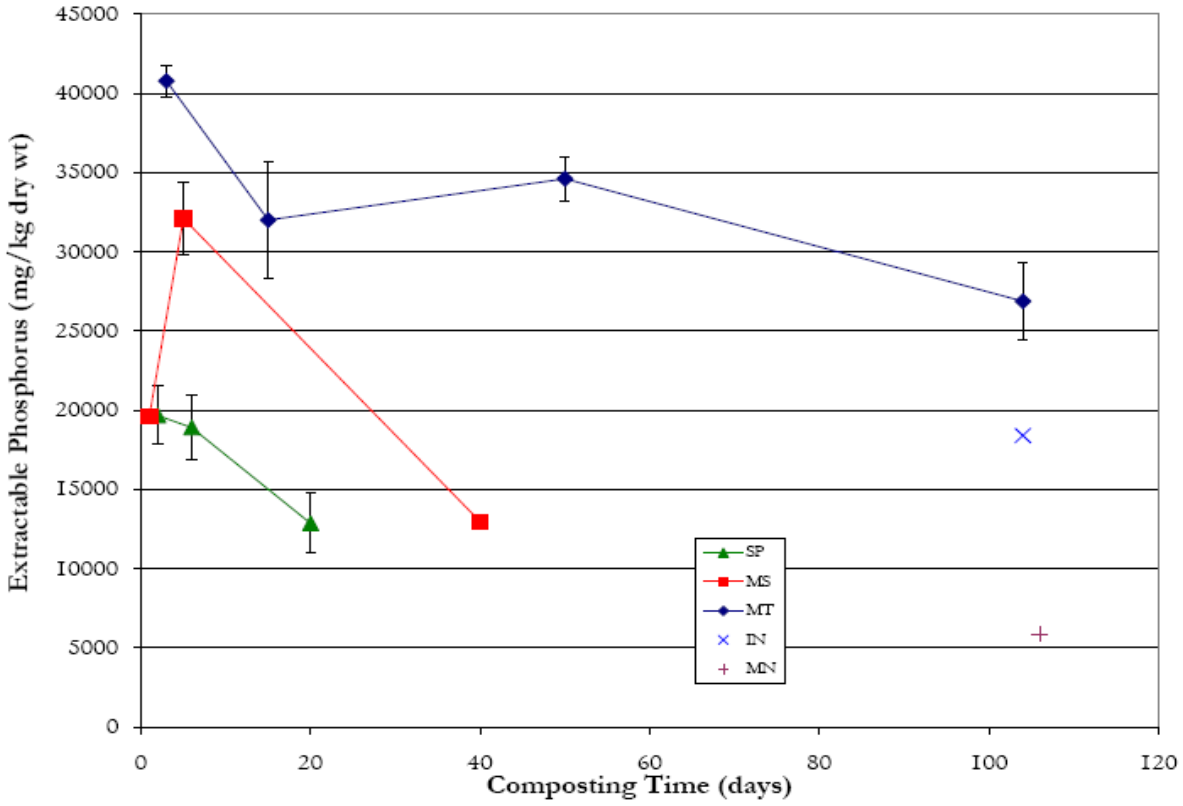


Figure 5.1-12: Mehlich 1 extractable phosphorus for treatments SP (▲), MS (■), MT (◆), IN (×) and MN (+) on dry weight bases. Initial sampling times may be offset for clarity. Error bars are 1 standard deviation.

Available phosphorus is a functional concept, and is loosely defined as the amount of phosphorus that would be available to plants during a growing season. This includes water soluble forms, portions of less soluble forms, such as calcium, aluminum and iron phosphates, that would solubilize via dissolution or desorption, and organic phosphorus that would undergo mineralization (Brady, 1990). Mehlich 1 extractable phosphorus is an operationally defined class of plant available phosphorus. Figure 5.1-12 shows that for all composts, phosphorus declined as composting time increased, which is similar to the results of Adler et al. (2003) who composted poultry litter. Chanyasak et al. (1983a) reported increasing available phosphorus with composting time, paralleling the trend for total phosphorus, which indicates that the increase was primarily dependent upon OM loss. Traoré et al. (1999) reported that the changes in available phosphorus were feedstock dependent, decreasing in a sewage sludge/wood chip mix, but increasing in a foodwaste compost. Due to increases in less readily extractable phosphate in both composts, Traoré et al. (1999) suggested that phosphate complexed with calcium, magnesium or iron, reducing the solubility of phosphorus. Additionally, they concluded that leaching of soluble phosphorus and OM losses would have contributed to genuine or apparent increases in available phosphorus. The slight increase in organic phosphorus indicated that immobilization was not a major phosphorus transformation pathway.

Phosphorus and phosphorus compounds are not volatile, and thus the decline during composting in the present study may be related to both leaching and formation of less soluble precipitates. The higher pH levels during composting (Figure 5.1-19) indicate that phosphorus would have tended to form less soluble, inorganic phosphate complexes, especially with calcium and magnesium (McBride, 1994). Conversely, organic matter decomposition is accompanied by the transformation of the more labile organic fraction to a more stable, humified fraction. The higher molecular weight, polymerized (humified) OM is better able to form chelates than the lower molecular weight, less polymerized labile fraction. Therefore, as composting progressed, the tendency for calcium and magnesium chelation would have increased, preventing phosphate complexation, and enhancing solubilization of extant phosphate precipitates (Stevenson, 1994). Additionally, the stable OM would have been more effective in competing for phosphate adsorption sites (Stevenson, 1994; Bhatti et al., 1998). Thus, the reduction in phosphate

complexation would have increased the leaching potential. Both organic and inorganic forms of phosphorus may leach from uncomposted and composted manures (Schwartz et al., 2005).

5.1.3.5 Copper and zinc

The data set for total copper concentrations was square root transformed to obtain more homogeneous residuals for statistical analysis. However, the data presented in Figure 5.1-13 are the untransformed values. The variables treatment and time had overall statistically significant effects (Pr_{MAX} = 0.001). The initial and final copper levels in SP were statistically different (Pr = 0.0002); for MS and MT, the concentrations were statistically equivalent (Pr_{MIN} = 0.698). Comparing final concentrations of all treatments, MT was statistically different from every other treatment (Pr_{MAX} = 0.0007); the other treatments were all statistically equivalent (Pr_{MIN} = 0.207).

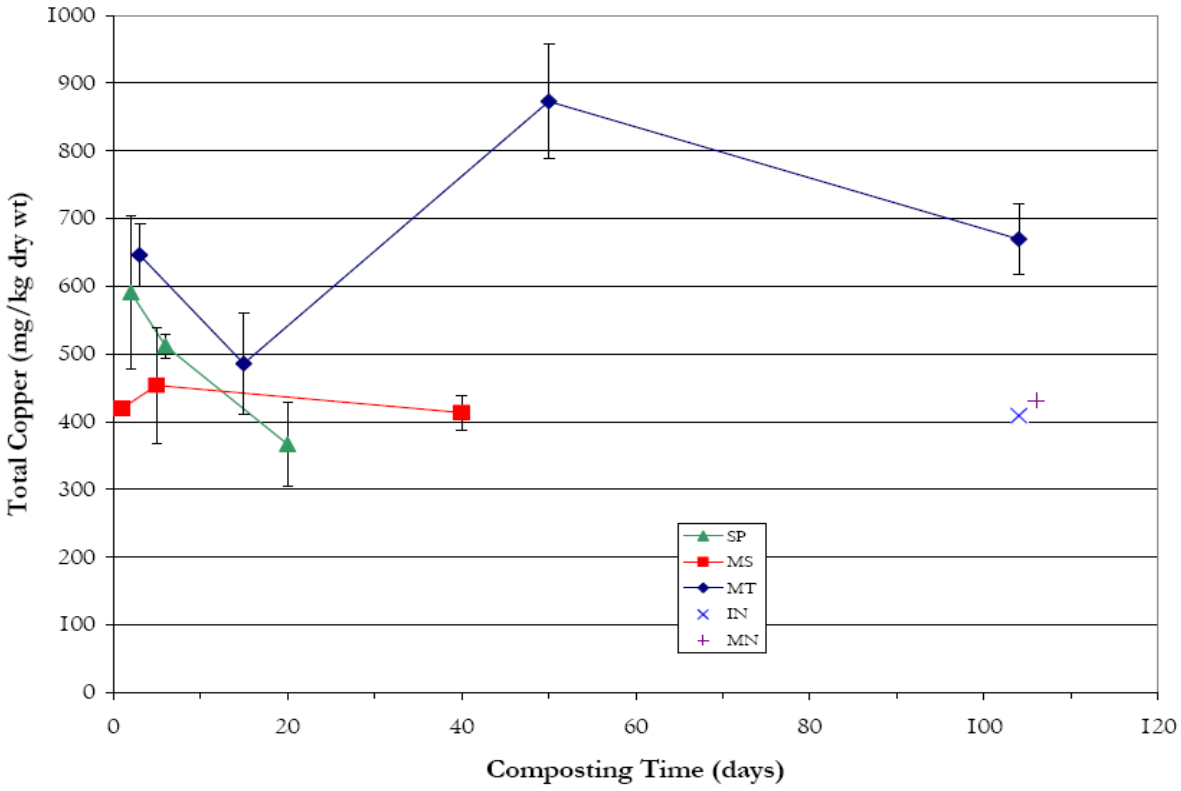


Figure 5.1-13: Total copper concentrations for SP (▲), MS (■), MT (◆), IN (×) and MN (+) on dry weight bases. Initial sampling times may be offset for clarity. Error bars are 1 standard deviation.

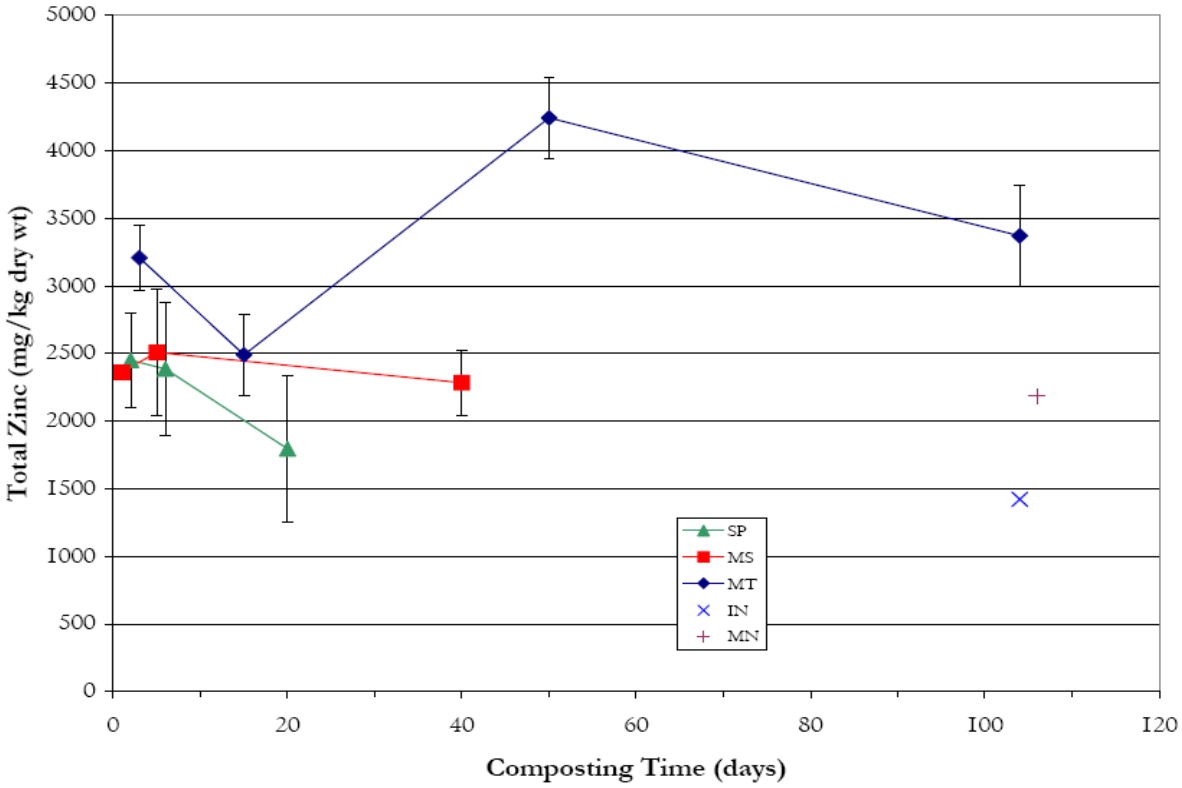


Figure 5.1-14: Total zinc concentrations for SP (▲), MS (■), MT (◆), IN (×) and MN (+) on dry weight bases. Initial sampling times may be offset for clarity. Error bars are 1 standard deviation.

For total zinc concentrations (Figure 5.1-14), the main effects, treatment and time, were statistically significant ($Pr_{MAX} = 0.005$). The initial and final concentrations of total zinc for MS and MT were statistically equivalent ($Pr_{MIN} = 0.590$); for SP, the levels were significantly different ($Pr = 0.039$). Comparing final concentrations for every treatment, the following were statistically equivalent ($Pr_{MIN} = 0.114$): IN-SP, MN-SP, MN-MS and SP-MS. All other pairwise comparisons were statistically different ($Pr_{MAX} = 0.048$).

The temporal changes in total (using concentrated acid for sample digestion) copper and zinc, respectively were very similar. For MT, there were net increases in copper and zinc (3.6 and 5.1%, respectively) as calculated between the initial and final concentrations, although the increases were not statistically significant ($\alpha = 0.05$). However, the concentrations oscillated

during composting. MS also showed concentration fluctuations during composting with final losses between initial and final concentrations of 3.4% for copper and 1.5% for zinc, although the increases were also not statistically significant. SP showed monotonic decreases for both copper and zinc, with final losses of 27% and 38% respectively, the decreases being statistically significant. The high losses for SP were not unusual, and were similar to MT for similar time periods. While SP was composted for 19 days, MT had a sampling point at 14 days, with losses of 24.9% and 22.4% for copper and zinc, respectively. MS did not have a corresponding sampling point for comparison.

The temporal fluctuations in copper and zinc concentration for all three composts appear to have been the net result of apparent increases due to OM loss and actual losses from leachate runoff (particulate or dissolved forms). With organic matter loss via decomposition (Figure 5.1-11), the concentrations of copper and zinc would have apparently increased, if no losses in leachate runoff had occurred. In the present study, leachate loss was not prevented and although the composting was conducted under a sheltered facility, water did collect within the composting area following intense rain, which would have facilitated nutrient losses.

Copper and zinc readily complex with organic and inorganic ligands, forming both readily extractable (eg, water extractable) and less soluble complexes (McBride et al., 1989; Stevenson, 1994; Hsu et al., 2001). Although these cations may form less soluble complexes reducing leachate loss, the presence of high amounts of organic matter may have enhanced metal solubility to a greater extent than the formation of less soluble complexes. As pH increases, copper and zinc tend to form less soluble complexes and adsorb onto mineral surfaces (McBride, 1989); the pHs of the composts were alkaline (Figure 5.1-19). However, in the presence of OM, dissolved organic matter (DOM) complex with these cations to form soluble DOM-metal moieties, reducing the formation of less soluble metal complexes; this occurs at acidic and alkaline conditions (McBride, 1989; Zhou et al. 2001; Karathanasis et al., 2005). Therefore, the high OM content of the composts may have been pivotal in enhancing the leachability of copper and zinc. Garcia et al. (1990) reported that total copper and zinc concentrations increased (generally non-monotonically) during the composting of a variety of feedstocks, but also indicated that leaching potentials were minimized by regulating the moisture content of the compost piles.

5.1.3.6 Phenols

Determinations of equivalent phenol concentrations (EPC) were based on a standard curve (Figure 5.1-15), which was obtained using 4-hydroxycinnamic acid (p-coumaric acid) as the reference material. A range of 100-500 mg/L was selected because 1) most of the aqueous extracts could be analyzed in the undiluted state, reducing potential sample preparation error, and 2) 500 mg/L was approaching the maximum detection limit of the spectrophotometer.

Recovery analysis was conducted for two phenolic substances. Recovery of coumaric acid was 97-98%; for phenolic acid it was 94-96%.

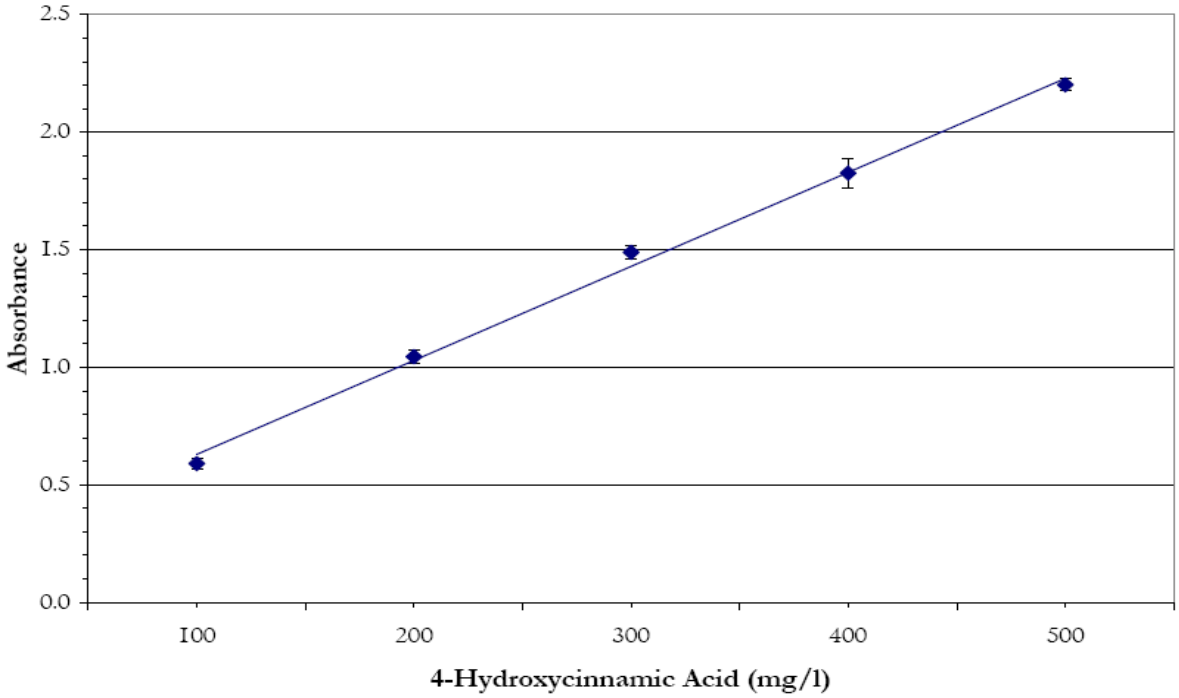


Figure 5.1-15: Standard (regression) curve for absorbance of 4-hydroxycinnamic acid. Error bars are 1 standard deviation.

Equivalent phenol concentrations (EPC) were calculated on both dry weight and ash-free, dry weight bases (Figure 5.1-16), although the statistical analysis was based on ash-free dry weight data. Both time and treatment effects were statistically significant ($Pr_{MAX} = 0.0001$). Time had an overall statistically significant effect on treatment ($Pr_{MAX} = 0.0001$), as well as on each

individual treatment (Pr_{MAX}= 0.005). Comparing initial and final concentrations, the levels for each compost (SP, MS and MT) were statistically different (Pr_{MAX} = 0.002). Comparing concentrations on the day of field application, IN was statistically equivalent to MN (Pr = 0.072) and MT (Pr = 0.185); all other concentrations were statistically different (Pr_{MAX} = 0.0002).

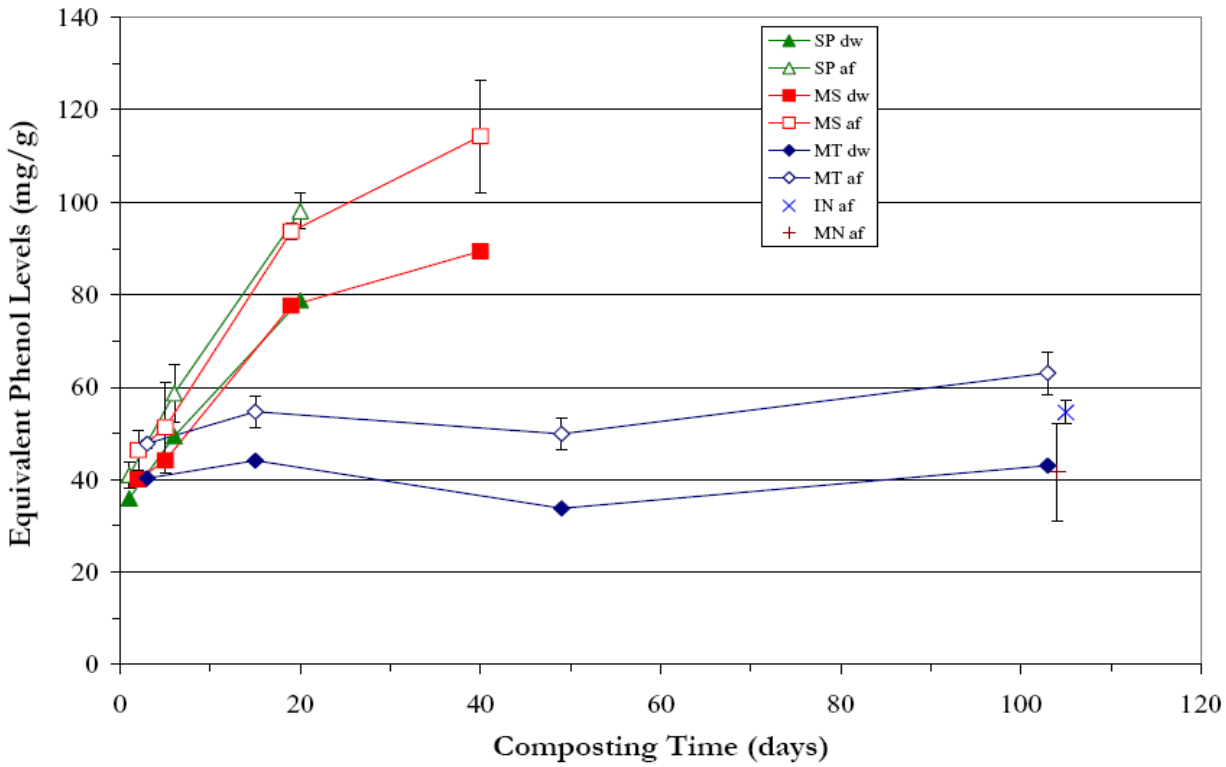


Figure 5.1-16: Equivalent phenol concentrations for treatments SP (▲), MS (■), MT (◆), IN (×) and MN (+). Solid symbols are on dry weight bases (dw). Open symbols, plus IN (×) and MN (+) are on ash free dry weight basis (af). Initial sampling times may be offset for clarity. Error bars, which are associated with the open symbols, are 1 standard deviation.

The phenolic acid concentrations, based on OM (ash free, dry weight) content, increased for all of the composts. The increase for SP and MS was more rapid than for MT, with the increases being partially due to the loss in organic matter. However, SP had the greatest rate of OM loss, while the losses for MS and MT were more similar (Figure 5.1-11) over the same

approximate time period of 15 – 20 days. The increases may also have been genuine, since as decomposition progresses, catabolic breakdown of OM may have caused the release of phenolic substances (Gallet et al., 1995); extracellular phenolic structures are also produced by microbes (Stevenson, 1994). The similar temporal profiles of SP and MS, and dissimilarity to MT may have been related to the different decomposition dynamics as reflected in the similar temperature profiles of SP and MS, which were different from MT (Figure 5.1-1).

Garcia et al. (1991a) and Liao et al. (1994) reported decreasing concentrations of water extractable phenols during the composting of a variety of feedstocks, although Zubillaga et al. (2006) reported statistically invariant concentrations during the co-composting of MSW sludge and sawdust. However, using a weak polar solvent (50% methanol solution), Erhart et. al. (1999) reported that phenol levels in a 50% biowaste/50% yardwaste compost increased over a period of 4 months, which was similar to the results in the present study. Some of the variability between the different investigations may have been due to disparate extraction methodologies, in addition to different feedstocks and composting conditions. Garcia et al. (1991a), Liao et al. (1994), and Zubillaga et al. (2006) all used cold water extractions. Autoclaving, the procedure used in the present study, is an aggressive water extraction technique and may have been more similar to the methanol extraction than to the cold water extraction methods.

In Figure 5.1-16, the EPC, which were calculated on a dry weight basis, were equivalent to a range of approximately 275 mg/L to 600 mg/L for all treatments. Marambe et al. (1992) reported that phenolic acid concentrations of 50 mg/L, significantly reduced sorghum seed germination in seed bioassay tests.

Although low, stable concentrations of phenols have been suggested as stability indices (Riffaldi et al., 1986; Keeling et al., 1994a; Liao et al., 1994), it appears that extraction procedures, feedstock characteristics and possibly composting conditions may affect levels of phenols. Riffaldi et al. (1986) cautioned about applying the results of their study universally (ie, all feedstocks). In the present study, the lack of stabilization of phenol levels over the long term (ie, compost MT) may have been due to the infrequent sampling points creating an apparent increase, rather than stabilization. Therefore, phenol concentrations, based on OM content, were considered as a stability index.

5.1.3.7 Fatty Acids

Concentrations of the fatty acids (C4-C18:3), shown in Figures 5.1-17 and 5.1-18, were determined using gas chromatography. Statistically, the variable treatment, was not significant (Pr = 0.066), while time had an overall significant effect (Pr_{MAX} = 0.0001). Both interaction terms, time*fatty acid and treatment*time*fatty acid, were statistically significant (Pr_{MAX} = 0.003). One degree of freedom contrasts indicated that differences between the initial and final concentrations for MS were significant (Pr = 0.007), but the differences between the initial and final concentrations for SP and MT were not significant (Pr_{MIN} = 0.091). There were no

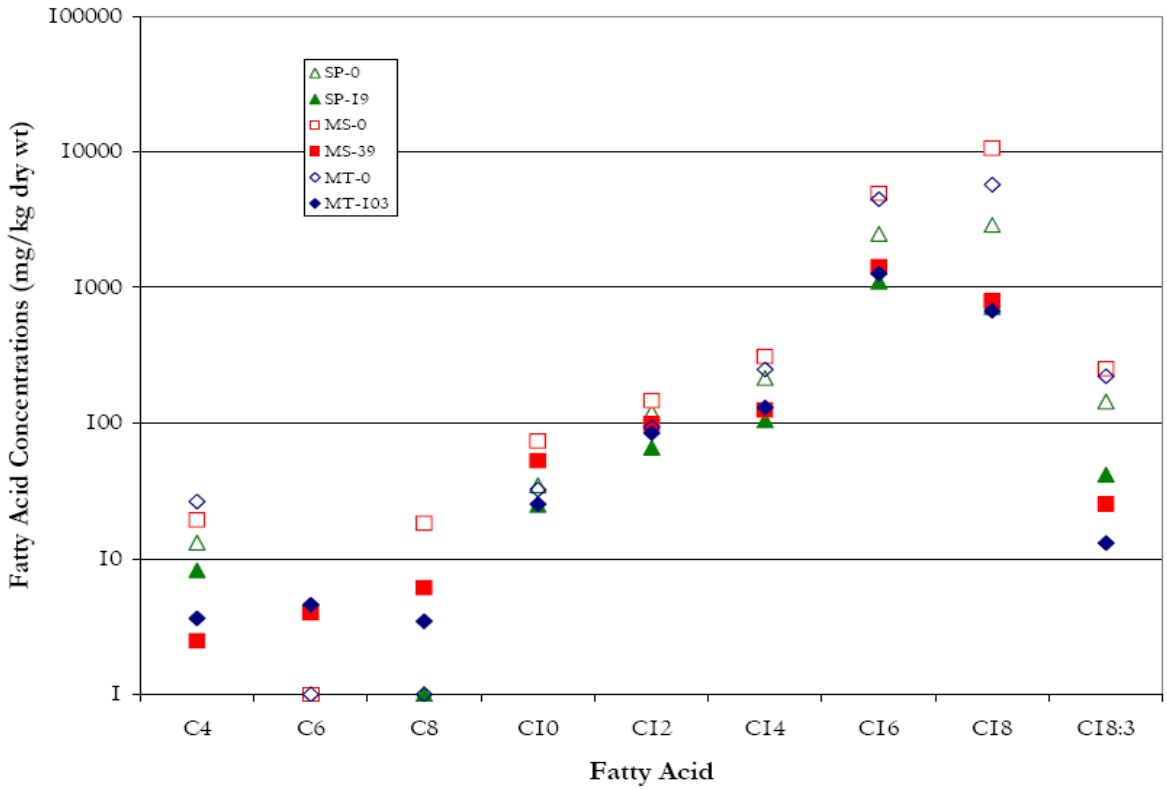


Figure 5.1-17: Fatty acid concentrations for composts SP (19 day, ▲), MS (39 day, ■), MT (103 day, ◆). Open symbols are concentrations of initial mixtures, and solid symbols represent final concentrations (on day of application). Numbers associated with the treatments, in legend, indicate days of composting. Fatty acids are coded for carbon chain length (C4 means a 4 carbon chain; C18:3 indicates 3 double bonds).

statistically significant differences between any of the final concentrations (Pr_{MIN} = 0.105) for all treatments (MN, IN, SP, MS and MT).

Figure 5.1-18 shows the effect of adding the bulking agent (wood chips/leaves) to the manure, on the fatty acids concentrations, and Figure 5.1-17 shows the changes that occurred during composting. Both Figures demonstrate that with the exception of butyric (C4) and linolenic (C18:3) acids, there was a general increase in concentrations as the molecular weights increased, which may partly be a reflection of the greater volatility as molecular weight decreases. The elevated levels of higher molecular weight fatty acids may also be due to the fact that the C12-C20 fatty acids are the major fatty acid types in plant leaf tissue and are generally, the most common bacterial fatty acids (Harwood et al., 1984).

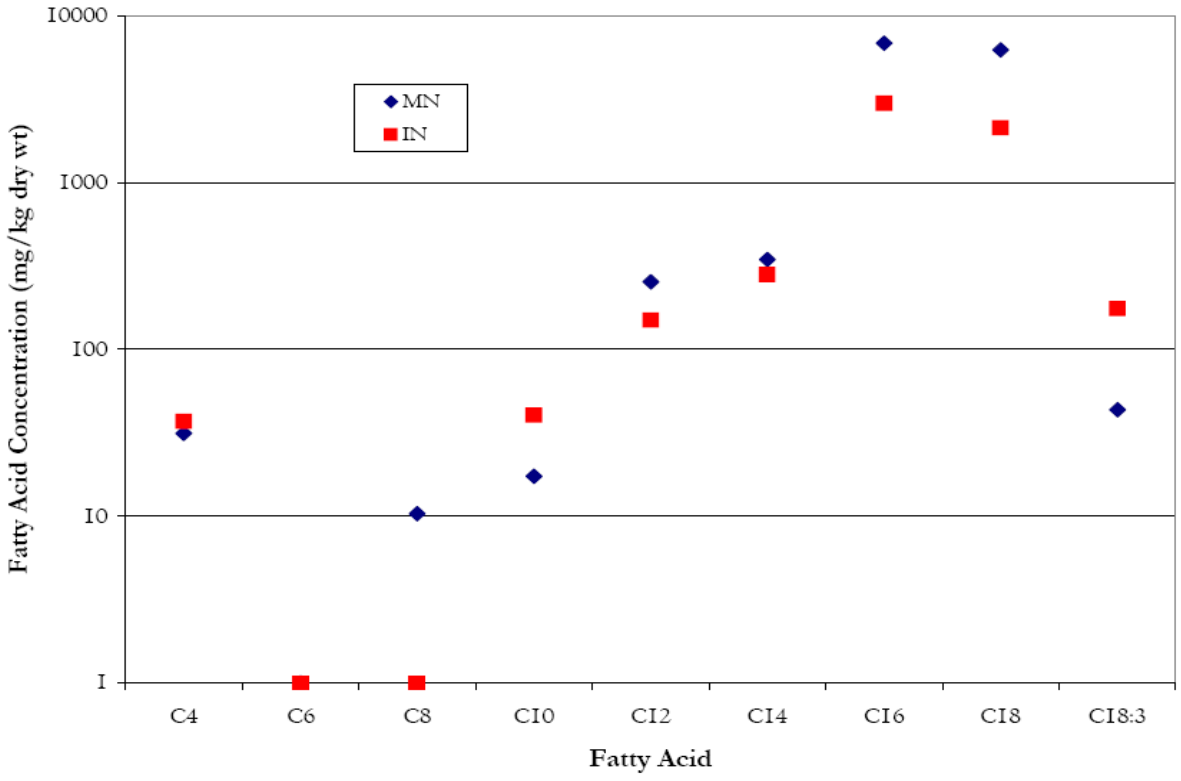


Figure 5.1-18: Fatty acid concentrations for MN (manure, ◆) and IN (uncomposted feedstock, ■). Fatty acids are coded for carbon chain length (C4 means a 4 carbon chain; C18:3 indicates 3 double bonds).

Acetic acid (C2) levels, determined using a titration method, are not indicated in the Figures. The concentrations for all of the samples, except for the final concentrations of SP13 and MT11, were less than 1000 mg acid/kg sample (wet weight), the detection limit. For SP13, the concentration was 1750 mg/kg; for MT11 the concentration was 1050 mg/kg. As the detection limit of the gas chromatograph was about three orders of magnitude lower than the titration method, an accurate relationship between the acetic acid and higher molecular weight fatty acids could not be determined.

Fatty acids are the building blocks for lipids and consequently lipid decomposition will release fatty acids. Anaerobic decomposition of carbohydrates also produces fatty acids, and as anoxic sites are inherent in composting piles (Atkinson et al., 1996), fatty acids will also be released via this mechanism. The increases in some of the individual fatty acids during composting, such as with C6 and C8, may have been due to the release of these during OM decomposition or the decomposition of higher molecular weight fatty acids.

Volatile fatty acid (C2-C6) concentrations generally decrease during composting, although not necessarily monotonically (Veeken et al., 2000; Ozores-Hampton et al., 2002). In the present study, the changes in levels of C6 and C8 showed inconsistency (Figure 5.1-17). Reductions in lower molecular weight fatty acids can be due to volatilization and losses in leachate; microbial catabolism will decrease concentrations of fatty acids of all molecular weights. There is a paucity of information concerning the trends of the higher molecular weight fatty acids during composting, although Marambe et al (1993) concluded that these fatty acids were more potent phytotoxins than those of lower molecular weight.

In the present study, treatment had no statistically significant effect ($\alpha = 0.05$). There were no statistical differences between the initial and final concentrations of either MS or MT, although for SP the initial and final concentrations were statistically significant. The lack of statistical differences between treatments may have been due to the fact that the range of concentrations spanned four orders of magnitude, overemphasizing the high concentrations and masking changes in the lower concentrations. This seems to be confirmed by the statistically significant effect of the interaction term, treatment*time*fatty acid. In addition, the coefficients of variation (COV) were very high in many instances (Table 5.1-1). The COV for MN and IN were calculated from duplicate samples. For SP, MS and MT, the COV were calculated from

three replicate piles; from each pile two duplicate samples were analyzed.

Due to the non-significance between the initial and final fatty acid concentrations for MS and MT, fatty acid was excluded as a potential stability index. Although the initial and final concentrations were statistically different for SP, the duration of composting was relatively short, and consequently more emphasis was given to MS and MT, which were composted for longer periods.

Table 5.1-1: Coefficients of variation for soil amendments, manure (MN) and composts (IN, SP, MS and MT). The letters, I and F, indicate initial and final compost mixtures, respectively. The zeroes in the data columns mean that the average fatty acid concentrations were zero.

	MN	**IN**	**SP – I**	**SP – F**	**MS – I**	**MS – F**	**MT – I**	**MT – F**
C4	0.14	0.33	1.14	0.88	1.73	1.73	0.53	1.73
C6	0.00	0.00	0.00	0.00	0.00	1.73	0.00	1.73
C8	1.41	0.00	0.00	0.00	1.73	1.12	0.00	1.73
C10	1.41	0.06	0.30	0.88	0.56	0.24	0.63	1.26
C12	0.38	0.07	0.05	0.43	0.65	0.17	0.38	0.35
C14	0.14	0.33	0.07	0.21	0.27	0.24	0.34	0.28
C16	0.02	0.00	0.75	0.50	0.73	0.21	0.39	0.24
C18	0.01	0.05	0.05	0.29	0.77	0.15	0.06	0.22
C18:3	0.10	0.16	0.77	1.13	0.28	0.28	0.06	0.92

5.1.4 Chemical Properties

5.1.4.1 pH

Figure 5.1-19 shows the change in pH during composting. Statistically, the main effects of treatment and time were significant (Pr_{MAX} = 0.0001), as was the interaction term treatment*time (Pr_{MAX} = 0.023). Time had a statistically significant effect on treatment pH for SP and MT (Pr_{MAX} = 0.0001), but not on MS (Pr = 0.775). However, due to non-monotonic temporal changes in pH, there were no statistically significant differences between the initial and

final pH levels of MS or MT (Pr_{MIN}= 0.648); the initial and final pH values for SP were statistically different (Pr = 0.0003). Comparing the final pH values for all treatments, MN was statistically equivalent to MS (Pr = 0.169) and SP was statistically equivalent to MT (Pr = 1.000). All other pairwise comparisons were statistically different (Pr_{MAX} = 0.01).

The pH values of all three composts were statistically invariant over time, which was the net effect of numerous potential mechanisms (see Chapter 2.3.2.5). Since microbial activity is optimum over a wide range of pH values, from about 5.6-8.4 (Jeris et al, 1978; Nakasaki et al., 1993), decomposition (ie, composting) readily progresses over this same pH range. Therefore, indexing compost stability to an "optimum" pH would not be feasible. However, a pH that levels off as composting progresses may indicate microbial activity has stabilized, and thus stable compost has been produced. This has been the basis for suggesting that a stable pH may be a

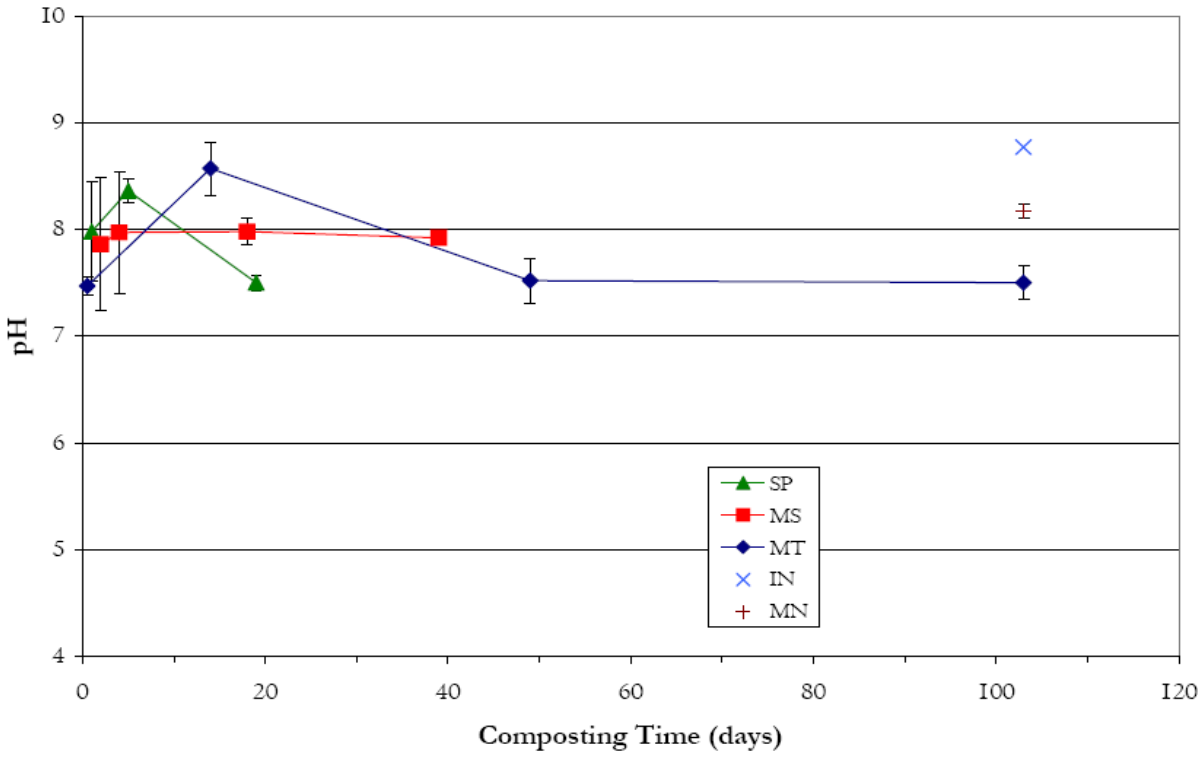


Figure 5.1-19: pH of soil amendments SP (▲), MS (■), MT (◆), IN (×) and MN (+). Initial sampling times may be offset for clarity. Error bars are 1 standard deviation.

complementary index for compost stability (Inbar et al., 1993; Avnimelech et al., 1996). However, because time did not have a statistically significant effect on all treatments and the initial and final pH values for each treatment were not necessarily statistically different, indicating that pH was generally invariant over time, pH would not be an appropriate indicator of compost progression.

5.1.4.2 Electrical Conductivity

The data set for electrical conductivity (EC) was log transformed to obtain more homogeneous residuals for statistical analysis. However, the data presented in Figure 5.1-20 are the untransformed values. The main effects of treatment and time were statistically significant

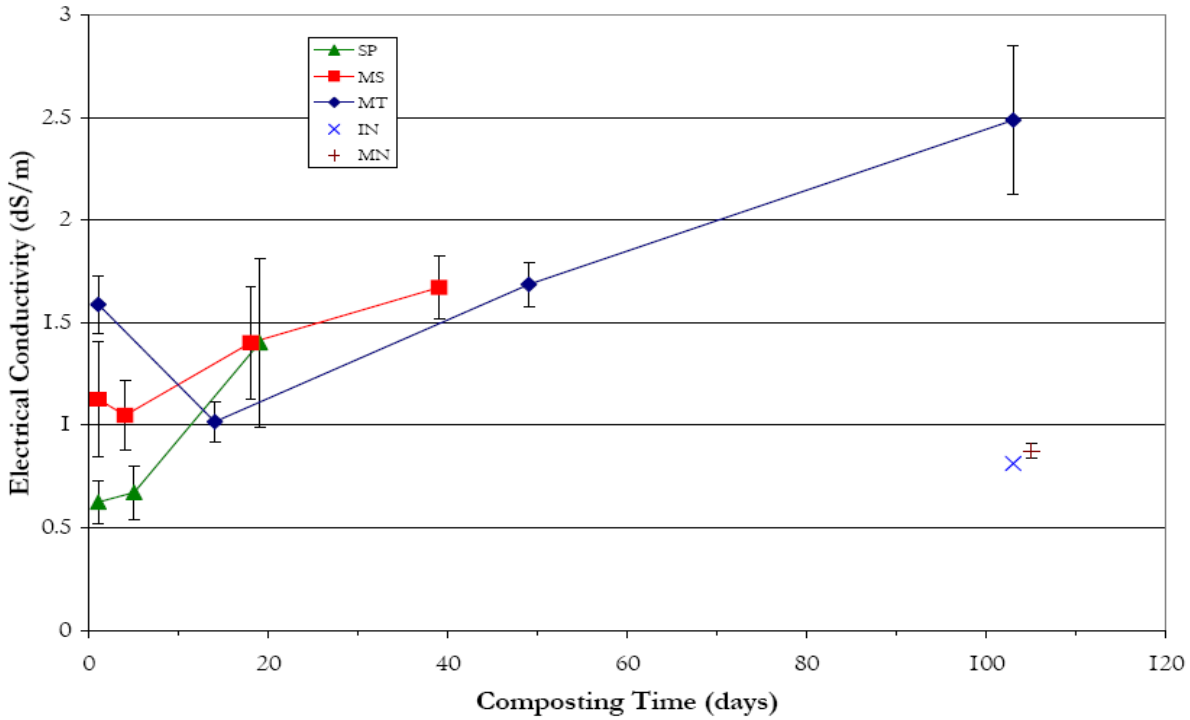


Figure 5.1-20: Electrical conductivity of soil amendments SP (▲), MS (■), MT (◆), IN (×) and MN (+). Treatments IN and MN are located at about 103 days to indicate the day of application, but are offset for clarity. Error bars are 1 standard deviation.

(Pr_{MAX} = 0.0001), as was the interaction term, treatment*time (Pr_{MAX} = 0.0001). The effect of time on each treatment was also statistically significant (Pr_{MAX} = 0.0001).

For each treatment (SP, MS and MT), the initial and final EC values were statistically different (Pr_{MAX} = 0.0001). Comparing the final values of all the treatments, MN and IN were statistically equivalent (Pr = 0.571); all other pairwise comparisons were statistically different Pr_{MAX} = 0.012).

The electrical conductivity increased with composting time indicating that OM loss may have been the predominant factor controlling the concentrations of ions that contributed to electrical conductivity. Chanyasak et al. (1983b) also reported increasing EC as composting progressed and measured concomitant increases in sodium levels, which indicates that a concentration effect of sodium, and probably other cations, were at least partially responsible for the increase in EC. Wang et al. (2004) reported increasing EC with composting time for two different cow manure composts, but decreasing EC with swine manure/wood compost.

Precipitation reactions (hypothesized by Avnimelech et al., 1996 in their study) and ion losses in leachate runoff (Eghball et al., 1997) could have contributed to net EC decreases or moderated increases. Wang et al. (2004) indicated that leaching "was avoided" during composting; leaching was not mentioned in the study of Chanyasak et al. (1983b).

Although a stable EC has been proposed as a complimentary composting index (Avnimelech et al., 1996; Wang et al., 2004), Figure 5.1-18 does not indicate stability. However, since it is possible that the lack of stability was the result of insufficient measuring points, EC will be included as a candidate composting index, even though this index is feedstock and/or composting condition dependent.

5.1.5 Calorimetry

5.1.5.1 Calorific Values

Gross calorific values based on ash free dry weights (Figure 5.1-21) were statistically analyzed. The main effects, treatment and time, were not statistically significant (Pr_{MIN} = 0.118). However, time did have a statistically significant effect on SP (Pr = 0.040), although not on MS or MT (Pr_{MIN} = 0.342). Comparing initial and final calorific values for SP, the increase of calorific values with time was statistically significant (Pr = 0.018), but for MS and MT, the

changes were not statistically significant (Pr_{MIN} = 0.094). Comparing the calorific values on the day of field application, the treatments were not necessarily statistically different (Appendix H).

Figure 5.1-22 indicates the calorific values, calculated on a dry weight basis, and provides insight into the decomposition dynamics occurring during composting. For the MT curve, the calorific values decreased with time, then stabilized with further composting. The calorific values became minima at about the same time the compost pile temperatures returned to mesophilic level (45^{O} C) at about 49 days (Figures 5.1-1 and 5.1-4). As OM decomposition rate is greater in the thermophilic range than the mesophilic range (Horwath et al., 1995; Sánchez–Monedero et al., 1999; Bidegain et al., 2000; García-Gómez et al., 2003 & 2005), the leveling off of the calorific values as the temperatures approached mesophilic levels, would correspond to a

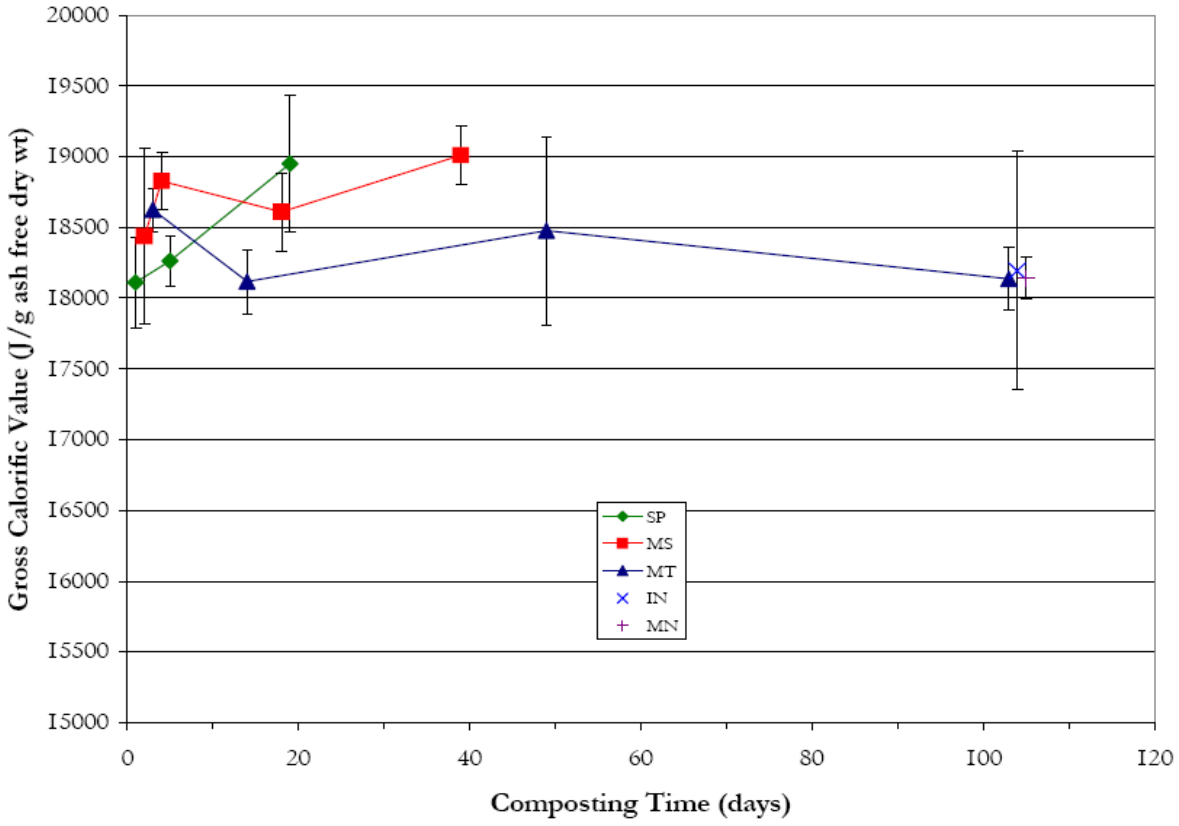


Figure 5.1-21: Gross calorific values on ash free, dry weight basis for SP (▲), MS (■), MT (◆), IN (×) and MN (+). Initial sampling times may be offset for clarity. Error bars are 1 standard deviation.

decrease in decomposition rate. Figure 5.1-5 confirms this as shown by the gradual decline in C:N ratio starting at about 7 weeks, compared to the more rapid reduction after about 1 1/2 - 2 weeks, which corresponds to the approximate time when the compost pile temperatures reached thermophilic level. The decrease in caloric values followed by a period of stabilization for MT is similar to the results reported by Vincelas-Akpa et al. (1994) for pruning wastes that were either aerobically decomposed or vermicomposted, and who attributed the trend to initial losses of organic matter juxtaposed with the later synthesis of more recalcitrant OM.

The MS curve followed the initial trend of the MT curve. While the SP curve showed an initial decrease followed by a period of stabilization, the initial and final calorific values were similar to the values of MS over the same time period. The specific pathways differed between the composts, which may have been due to the compositional differences (Sánchez–Monedero et al., 1999; Plaza et al., 2005), and composting conditions (Parkinson et al., 2004; Tiquia et al., 2005). Although the bulking agents for the composts were the same, the proportions varied, with an increasingly greater proportion of wood chips in the order MT > MS > SP.

In Figure 5.1-21 the MS and MT curves were invariant with time, while SP showed a statistically significant increase. The statistical invariance of MS and MT may have been due to two opposing decomposition processes: catabolism of OM and anabolism of more refractory components. When caloric values are calculated on an organic matter basis, the values are related to the recalcitrance of the OM. As decomposition progresses, the labile organic components are consumed prior to the more recalcitrant ones, and labile forms are also transformed into more refractory ones (Wilson, 1985; Lähdesmäki et al., 1988; Collins et al., 1990; Bernal et al., 1992; Tachimoto et al., 1995; Adani et al., 1995; Hsu et al., 1999).

Lignin is an organic matter fraction that is not readily biodegradable compared to other organic components (Wilson, 1985). Swine manure, wood and leaves all contain lignin. Lignin is one of the primary determinants for gross calorific values of wood (White, 1987; Rhén, 2004).

Although the dynamics of humic substance formation are not clearly understood, humic substances are partially derived from lignin-type structures/lignin degradation byproducts (Haider, 1994; Stevenson, 1994). Humic and fulvic acids have many similar structural characteristics as lignin (Inbar et al., 1990; Haider, 1994; Ramunni et al., 1994; Almendros et al.,

1999), and therefore, the energetic values may be similar (the present author's hypothesis). As composting progresses, the amount of humified OM generally increases (Miikki et al., 1994; Ouatmane et al., 2000; Schnitzer et al., 2000), and as the degree of humification increases, the calorific levels generally increase (Salmi, 1954; Lähdesmäki et al., 1988). Thus, the invariance in calorific values of MS and MT (Figure 5.1-21) may have been due to the degradation of lignin, being offset by the synthesis of recalcitrant humic substances. However, while lignin degradation appears to occur primarily during thermophilic phase (Horwath et al., 1996a; Sánchez–Monedero et al., 1999; García-Gómez et al., 2003 & 2005) the literature is less clear on when humification is predominant (Riffaldi et al., 1986; Chefetz et al., 1996; Hsu et al., 1999; Veeken et al., 2000). The similarity of MS and MT, but dissimilarity to SP, may be due to the fact that. MS and MT were processed through a full thermophilic phase, while SP was not. However, the invariances determined for MS and MT may also have been due to high sample variability (eg, MS at day 1 and MT at day 49), preventing statistical differences from being detected: for MS.

5.1.5.2 Stability Index

Calorific values were calculated based on both dry weight and ash-free dry weight. The temporal trends for calorific values based on dry weight (Figure 5.1-22) were very similar to the temporal trends of organic matter decomposition (Figure 5.1-11, reproduced above). There is an inverse relationship between caloric values (dry wt) and organic matter content or time of OM degradation (Kucera et al., 1967; Rsyzkowski et al., 1975; Bieńkowski, 1981; Zanoni et al., 1982; Hegazy, 1998). Therefore, there is no advantage to using calorific values based on dry weight as a stability index. It would be much simpler, quicker and cost effective to determine OM content.

Based on ash free weight, there was a general invariance of calorific values during the composting process (Figure 5.1-21). Statistically, the calorific values were invariant ($\alpha = 0.05$) for MS and MT, and there were no statistical differences between the initial and final values. Therefore, calorific values do not appear to be an appropriate composting index for swine waste/wood-leaf compost.

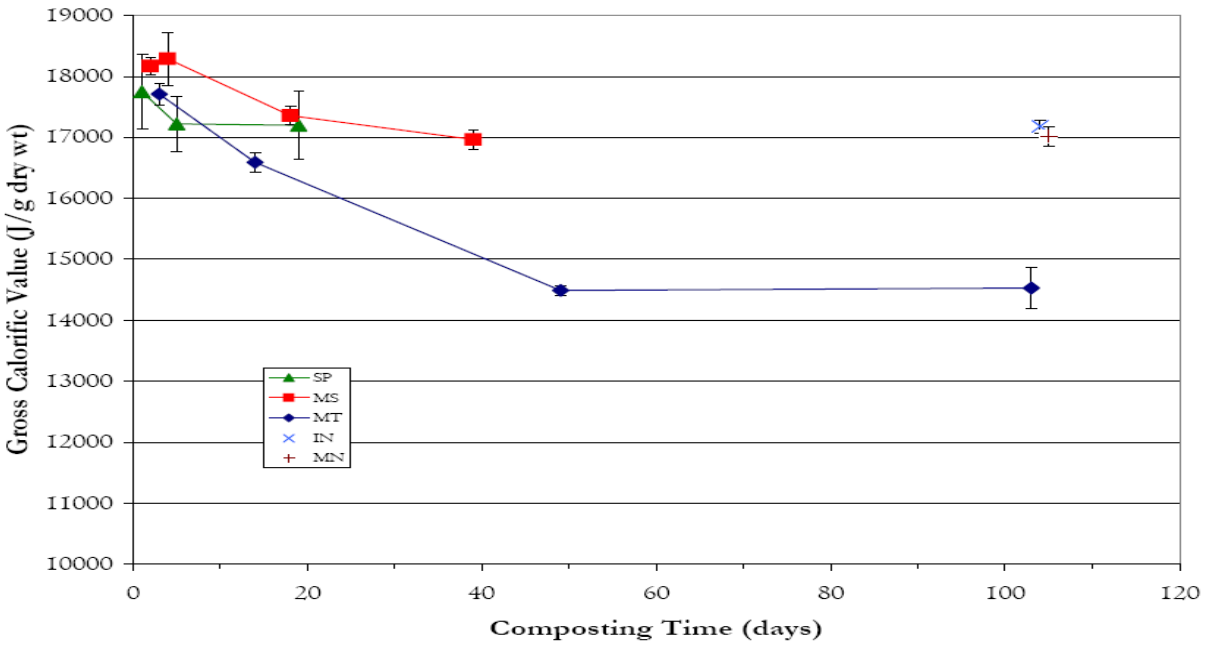


Figure 5.1-22: Gross calorific values on sample dry weight basis. Treatments are SP (▲), MS (■), MT (◆), IN (×) and MN (+). Initial times are offset for clarity only. Error bars are one standard deviation.

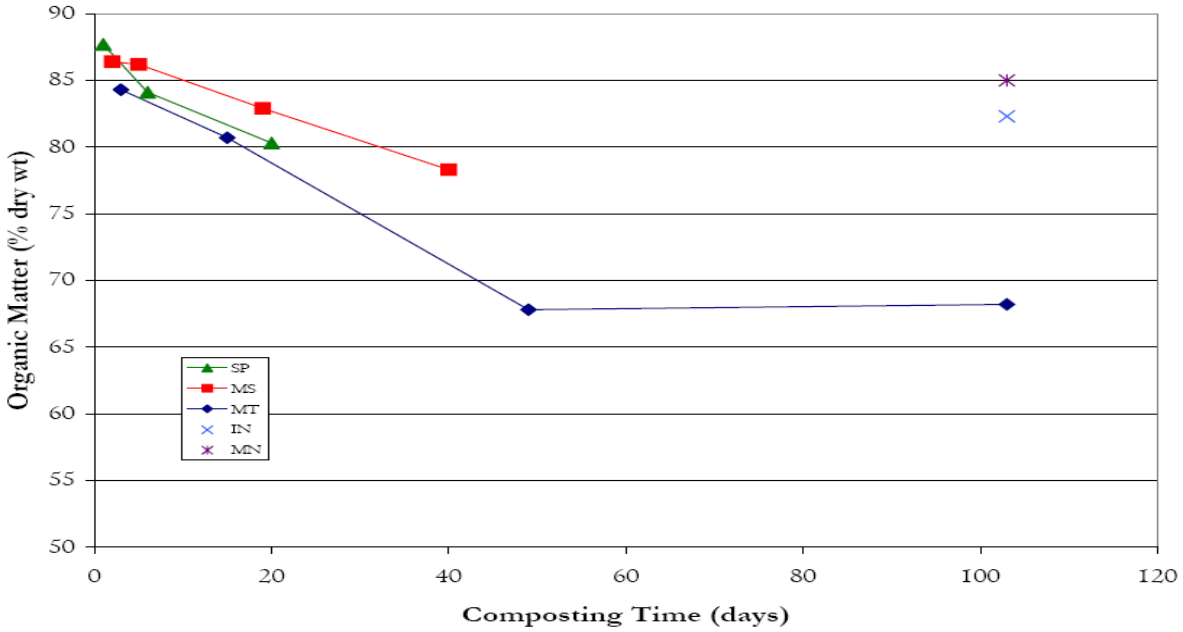


Figure 5.1-11: Organic matter content in organic amendments SP (▲), MS (■), MT (◆), IN (×) and MN (+). Initial sampling times may be offset for clarity. Error bars are 1 standard deviation.

5.1.6 Numerical Stability Indices

Numerical values have been proposed as thresholds for several stability indices. Since compost MT was the only one that was processed with the objective of achieving stability, it is possible to judge whether the compost was stable by comparing its characteristics to the threshold values.

Iglesias-Jiménez et al. (1992) proposed a C:N ratio of less than 12 as a necessary, but insufficient condition to ensure a "good degree of maturity". This conditional acceptance was proffered because 1) although the ratio of 12 would be similar to "well-humified soils", the authors acknowledged that the value for "well humified soils" can vary, 2) the different amounts of recalcitrant carbon and nitrogen in the feedstocks results in varying degrees of bioavailability and the carbon and nitrogen levels do not indicate potential biodegradability and 3) feedstocks with initially low C:N ratios may confound the index. In the present study, the final C:N for MT was 11.0. As the initial C:N of the manure for MT was 17.4, it does not appear that the C:N of the manure would have been a confounding factor for this index.

For nitrogenous species, Zucconi et al. (1987) proposed a maximum concentration for total ammoniacal nitrogen (TAN) (Figure 5.1-8) of 400 mg/kg, while a maximum of 1000 mg/kg was recommended by Tiquia et al. (1998). For "mature" compost, Forster et al. (1993)

Table 5.1-2: Stability indices for compost MT

INDEX	COMPOST MT	PROPOSED
C:N	11.0	< 12
TAN	526 mg/kg[1] (770 mg/kg)[2]	$\leq$ 400 mg/kg[1] $\leq$ 1000 mg/kg[1]
$NO_3 - N$	5500 mg/kg [1] (8000 mg/kg)[2]	> 300 mg/kg[1]
TAN/NO3	0.10	$\leq$ 0.16

1. dry weight basis
2. ash free dry weight basis

used a standard of greater than 300 mg NO_3-N/kg (Figure 5.1-9) as recommended by the Austrian Institute of Standards. Bernal et al. (1998b) proposed an TAN/NO_3 ratio of 0.16 maximum (Figure 5.1-10).

Table 5.1-2 lists the suggested stability indices with the values for compost MT at the time of application (103 days of composting). All of the proposed numerical indices, except possibly total ammoniacal nitrogen, indicate that the compost was stable.

5.2 Nitrogen Mineralization Study

5.2.1 Overview

To assess the nitrogen mineralization potential of the organic amendments (manure and composts), inorganic nitrogen levels and percent mineralized nitrogen were calculated at nine sampling points during the incubation study. Treatment (amendment) effects were compared at the end of the experiment (16 weeks) as this time period approximated the growing season in the field study. The mineralization rates at 16 weeks could then be related to the projected amendment mineralization rates in the field study.

Nitrate and total ammoniacal nitrogen concentrations were measured, although no statistical analyses were performed as inorganic nitrogen is comprised of both. Total nitrogen and C:N ratios were determined at the beginning and end of the experiment and the effects of treatment were statistically analyzed.

5.2.2 Results

5.2.2.1 Inorganic Nitrogen

Total inorganic nitrogen (Figure 5.2-1), total nitrate-nitrogen (Figure 5.2-2) and total ammoniacal nitrogen (TAN) (Figure 5.2-3) were calculated as the total amounts measured in the soil and soil/amendment mixtures (equation 4-2). Total inorganic nitrogen is deemed to be "plant available nitrogen" (PAN). Net concentrations of inorganic nitrogen (net PAN) (Figure 5.2-4), net nitrate-nitrogen (Figure 5.2-5) and net TAN (Figure 5.2-6) were calculated by subtracting the concentrations in the control soil (S) from the measured values (equation 4-3). Percent mineralized nitrogen (Figure 5.2-7), based on the initial amount of organic nitrogen, was

calculated accounting for the concentrations of inorganic nitrogen in the soil and the initial levels in the amendment (equation 4-4).

As incubation time increased the levels of inorganic nitrogen in the soil and soil/amendment mixes increased, which is indicated in Figures 5.2-1 and 5.2-4 (net inorganic nitrogen) and 5.2-7 (percent mineralized nitrogen). For net inorganic nitrogen and percent mineralized nitrogen data sets, statistical analyses indicated that the main effects, treatment and time, were statistically significant (Pr_{MAX} = 0.0001). Treatments were statistically compared at the end of the 16 week incubation with the results denoted in both Figures.

5.2.2.2 Carbon to Nitrogen Ratio

The data for C:N was log transformed to obtain more homogeneous residuals for statistical analysis. However, the untransformed data is presented (Figure 5.2-8). The main effects, treatment and time, and the interaction term, trt*time, were statistically significant (Pr_{MAX} = 0.0001). For each treatment, except the unamended control soil (S), the initial C:N ratio was statistically greater than the final C:N ratio (Pr_{MAX} = 0.0001). The initial and final C:N ratio for the soil were statistically equivalent (Pr = 0.592).

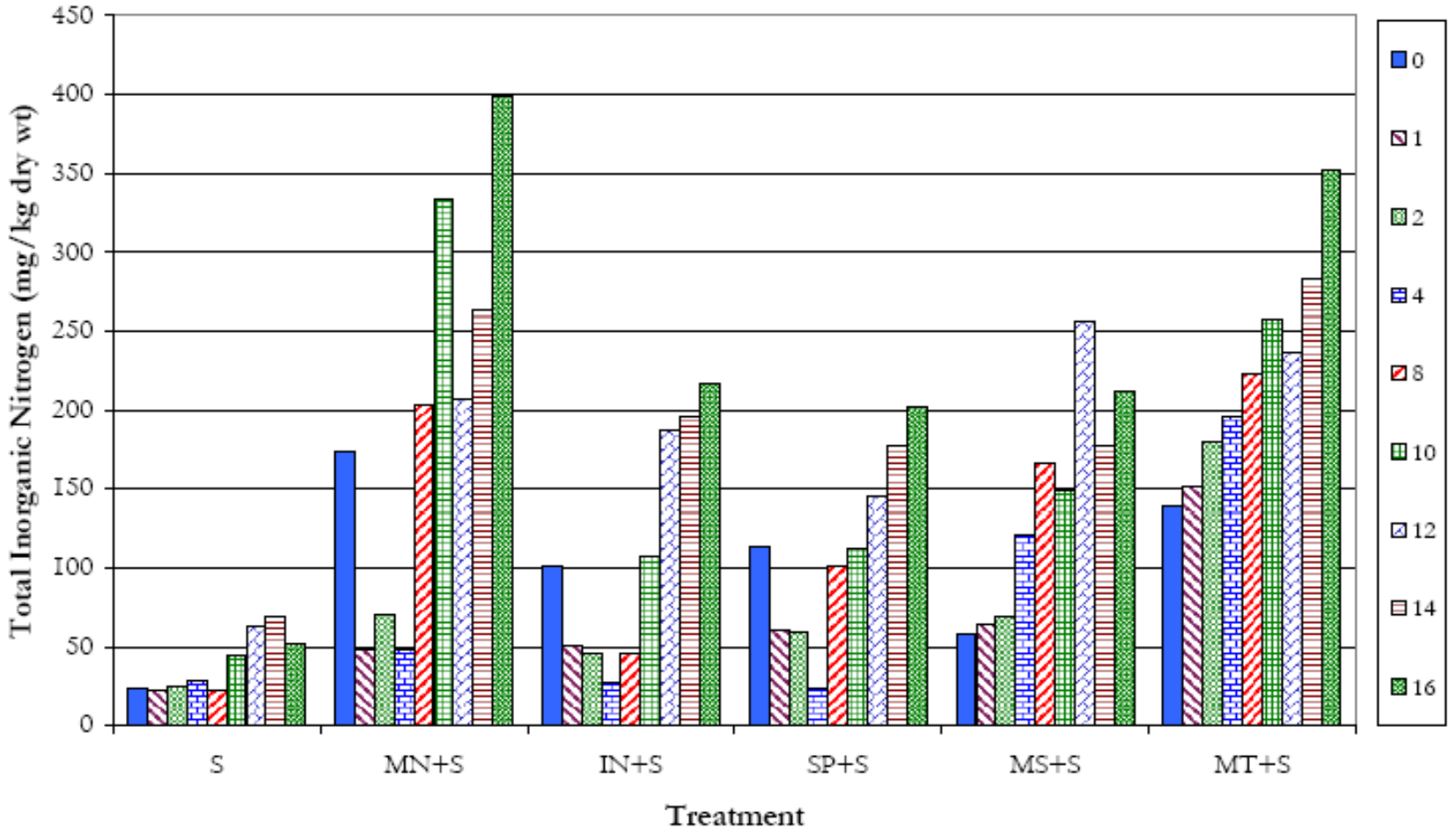


Figure 5.2-1: Total inorganic nitrogen (nitrate, nitrite and total ammoniacal nitrogen). Amendments (MN, IN, SP, MS and MT) were mixed with soil (S). Time legend is in weeks.

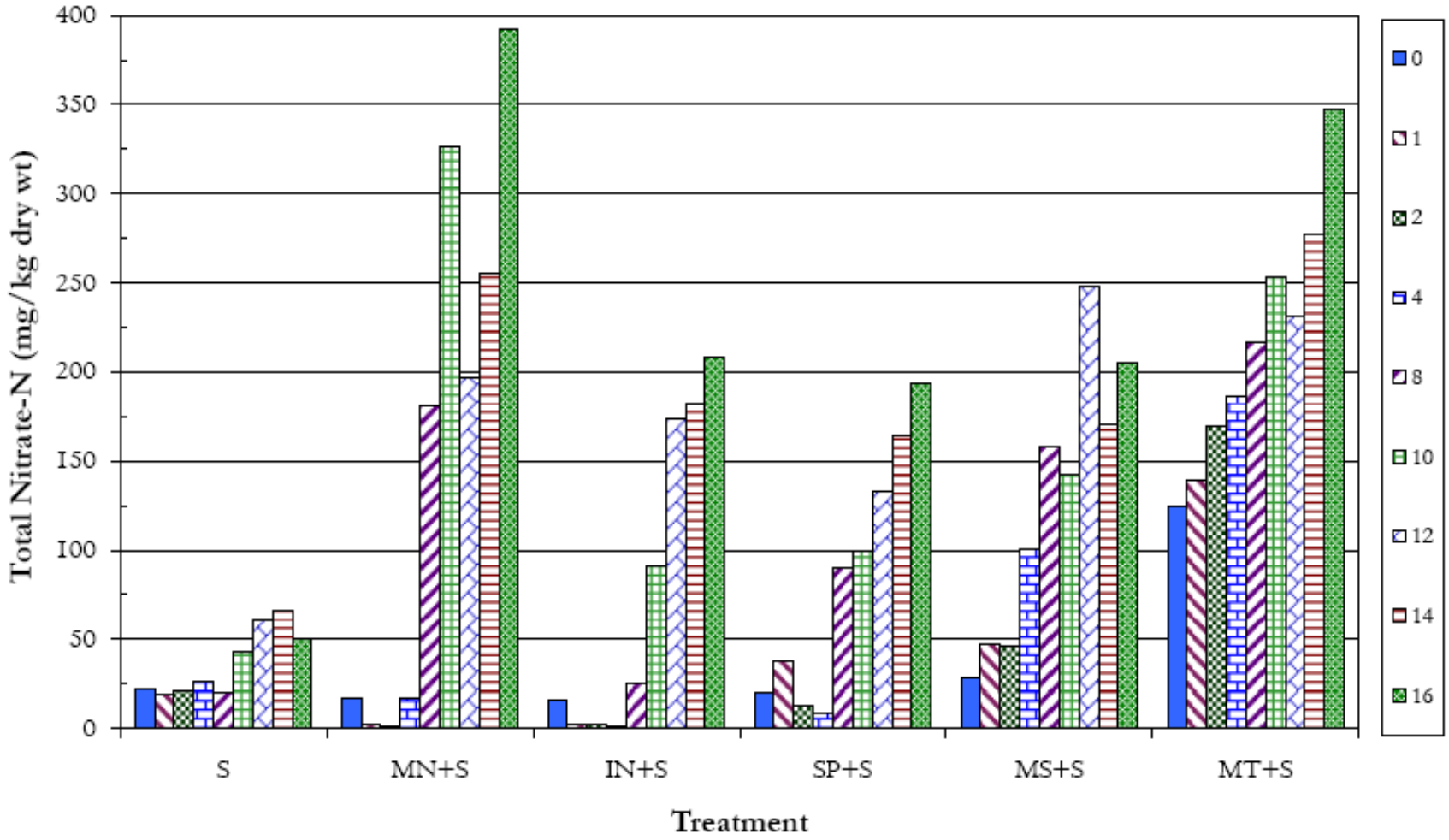


Figure 5.2-2: Total nitrate-nitrogen. Amendments (MN, IN, SP, MS and MT) were mixed with soil (S). Time legend is in weeks.

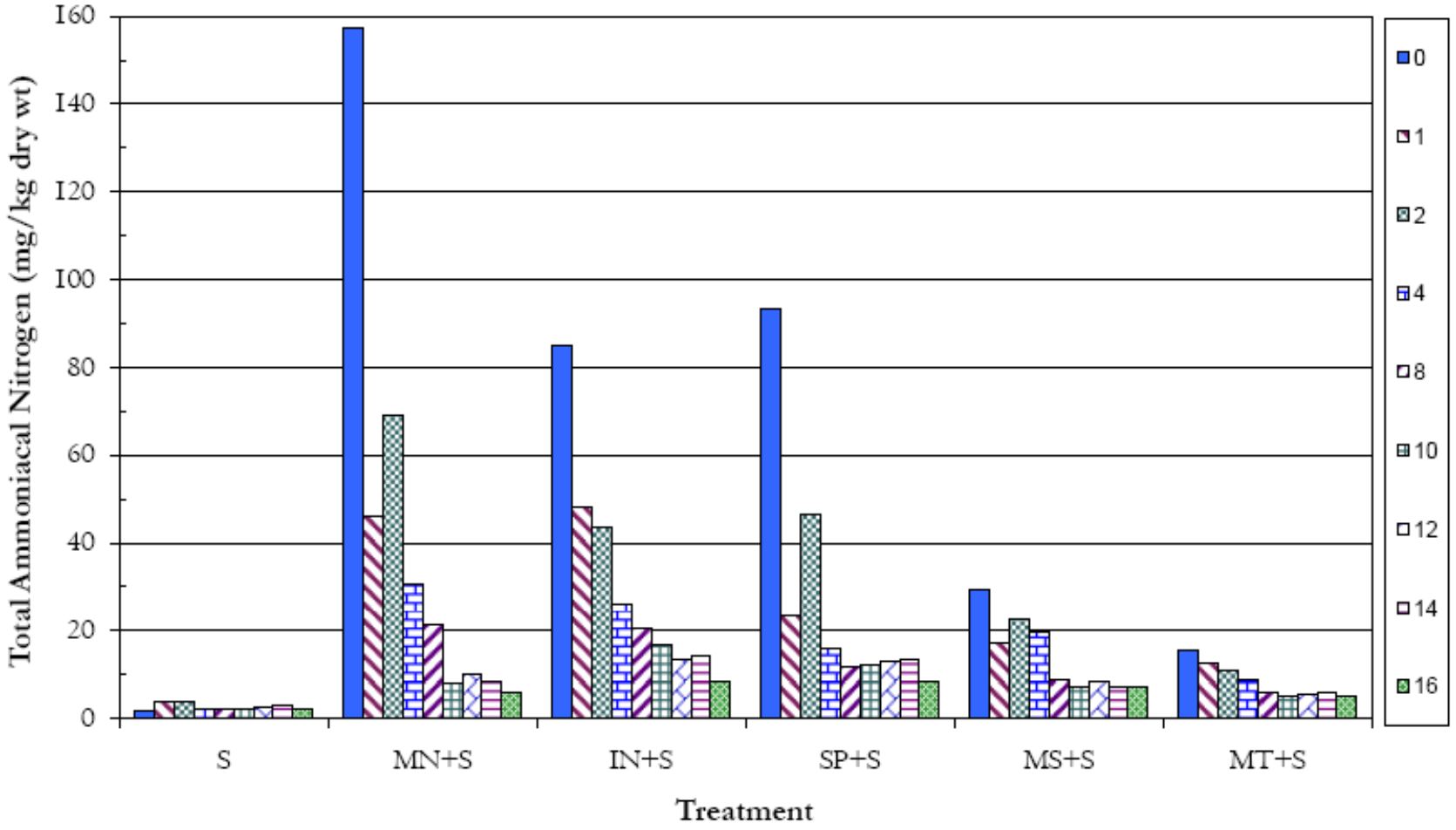


Figure 5.2-3: Total ammoniacal nitrogen. Amendments (MN, IN, SP, MS and MT) were mixed with soil (S). Time legend is in weeks.

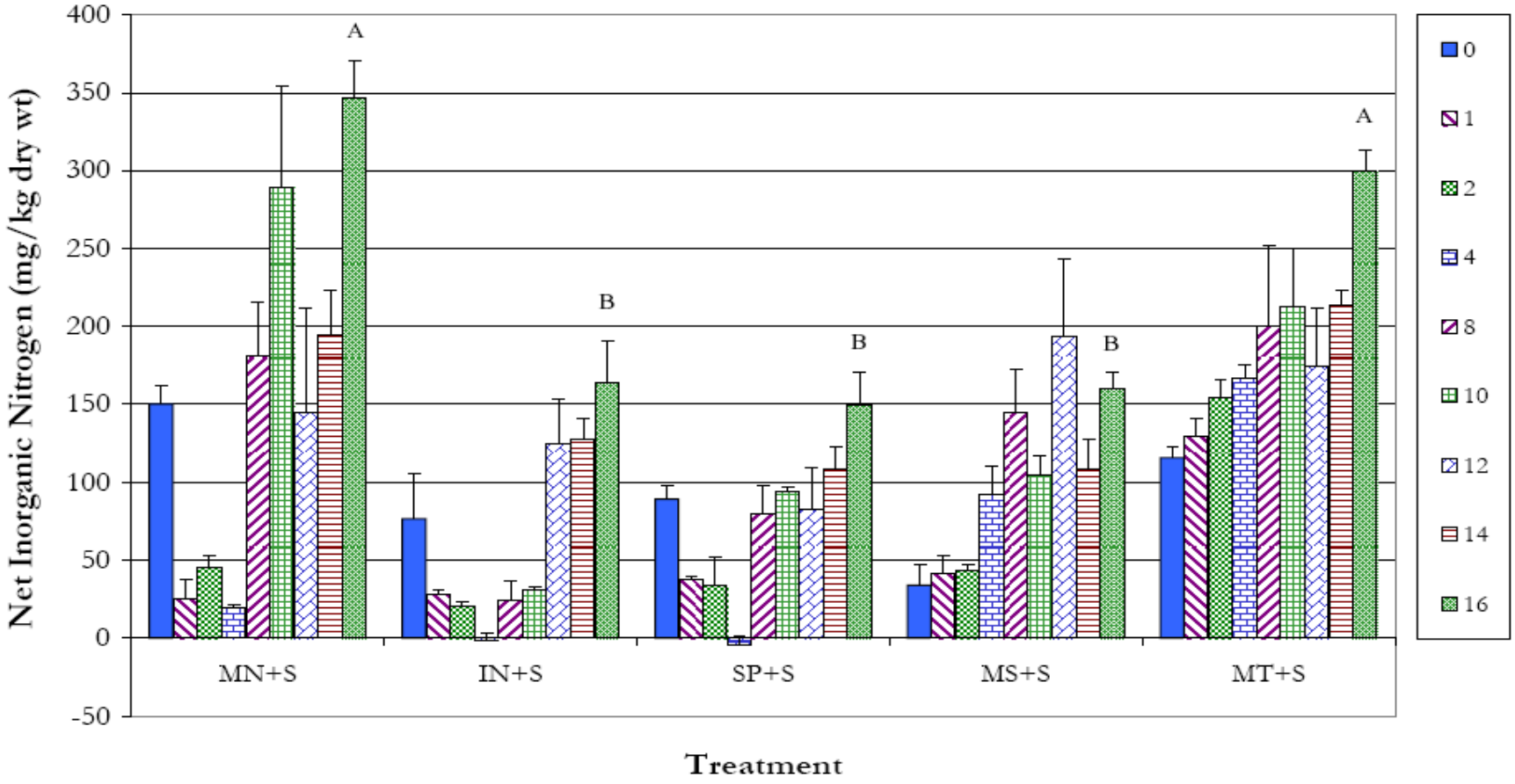


Figure 5.2-4: Net inorganic nitrogen. Soil (S) was mixed with the amendments: MN (manure), IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day). Time legend is in weeks. Treatments are compared at 16 weeks, and treatments with the same letter are statistically equivalent ($\alpha = 0.05$). Error bars are 1 standard deviation.

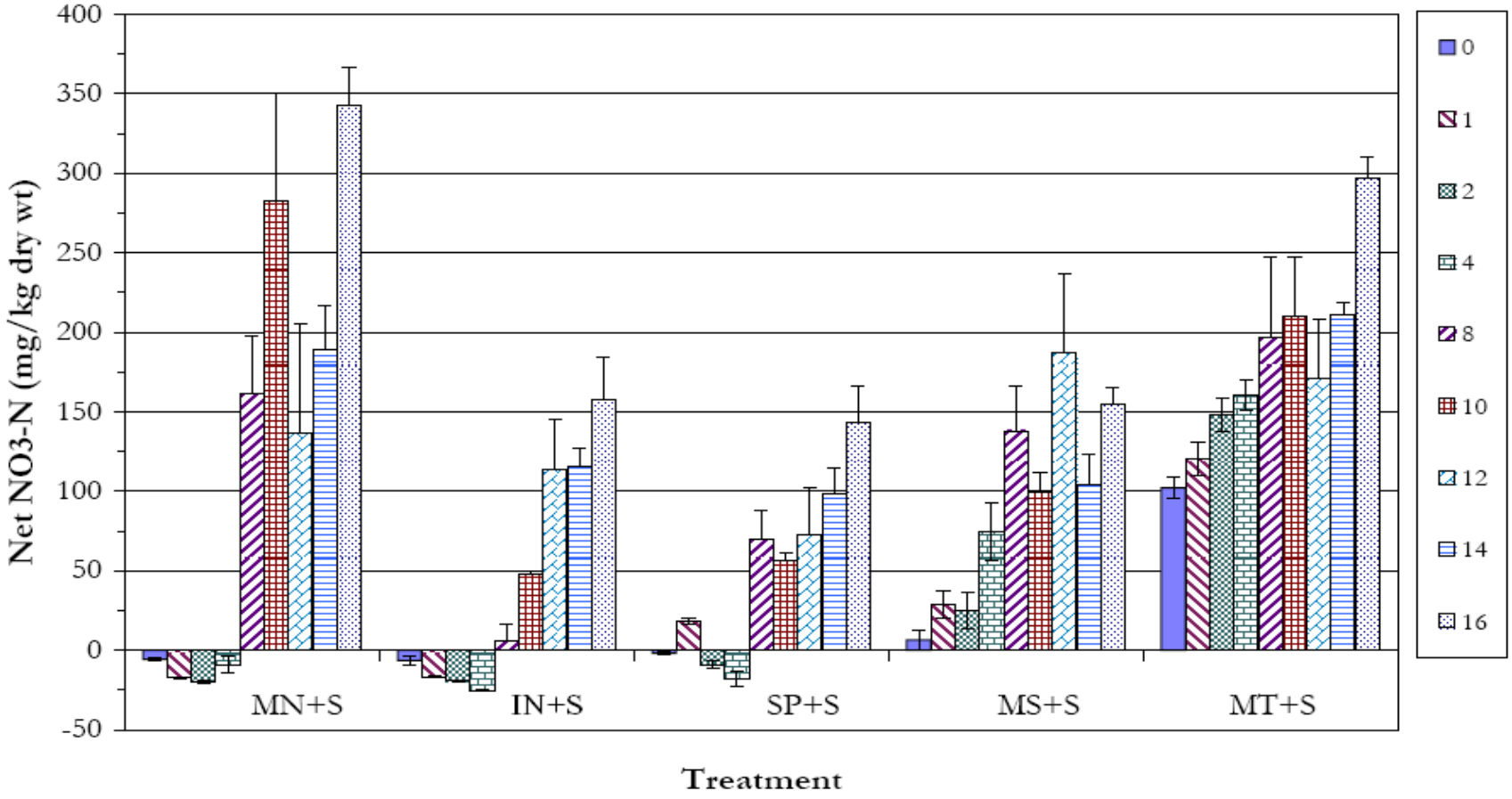


Figure 5.2-5: Net nitrate-nitrogen. Calculated as the difference between the concentrations of nitrate/nitrite in the amendment/soil mix and the control soil. Amendments (MN, IN, SP, MS and MT) were mixed with soil (S). Time legend is in weeks. Error bars are 1 standard deviation.

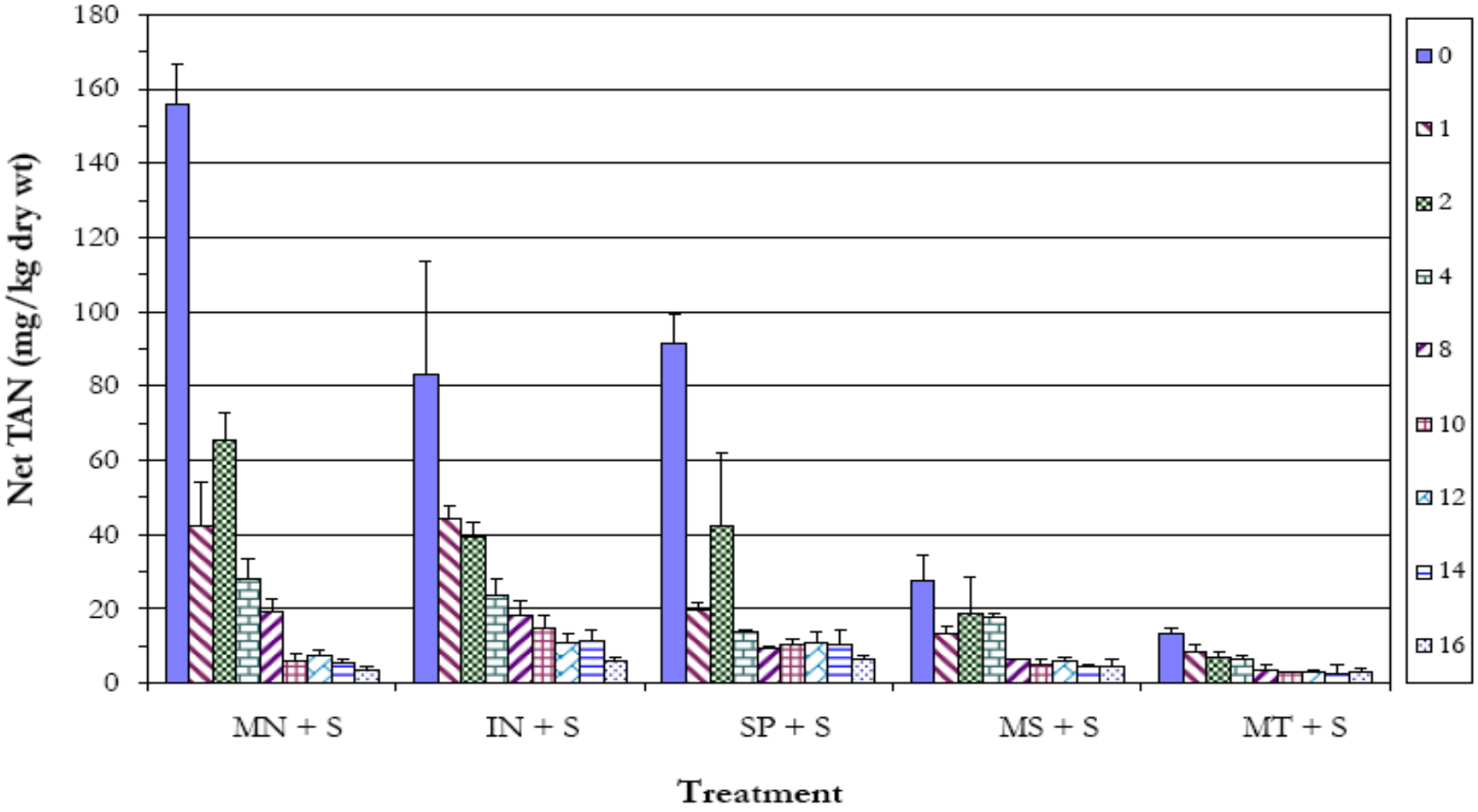


Figure 5.2-6: Net total ammoniacal nitrogen (TAN). Calculated as the difference between the concentrations of TAN in the amendment/soil mix and the control soil**.** Amendments (MN, IN, SP, MS and MT) were mixed with soil (S). Time legend is in weeks. Error bars are 1 standard deviation.

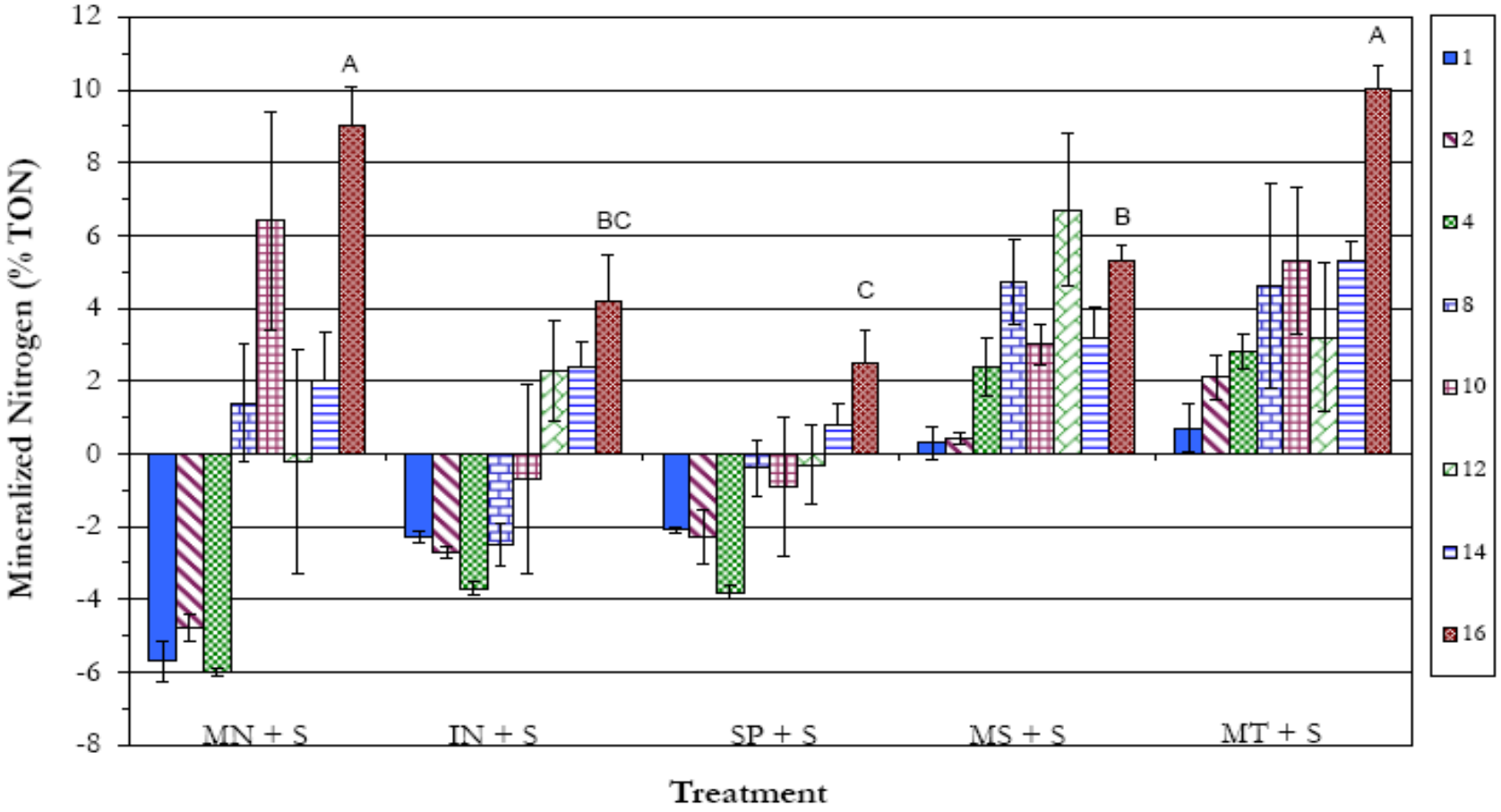


Figure 5.2-7: Mineralized inorganic nitrogen as a percentage of total organic nitrogen. Amendments (MN, IN, SP, MS and MT) were mixed with soil (S). Time legend is in weeks. Treatments are compared at 16 weeks, and treatments with the same letter are statistically equivalent ($\alpha = 0.05$). Error bars are 1 standard deviation.

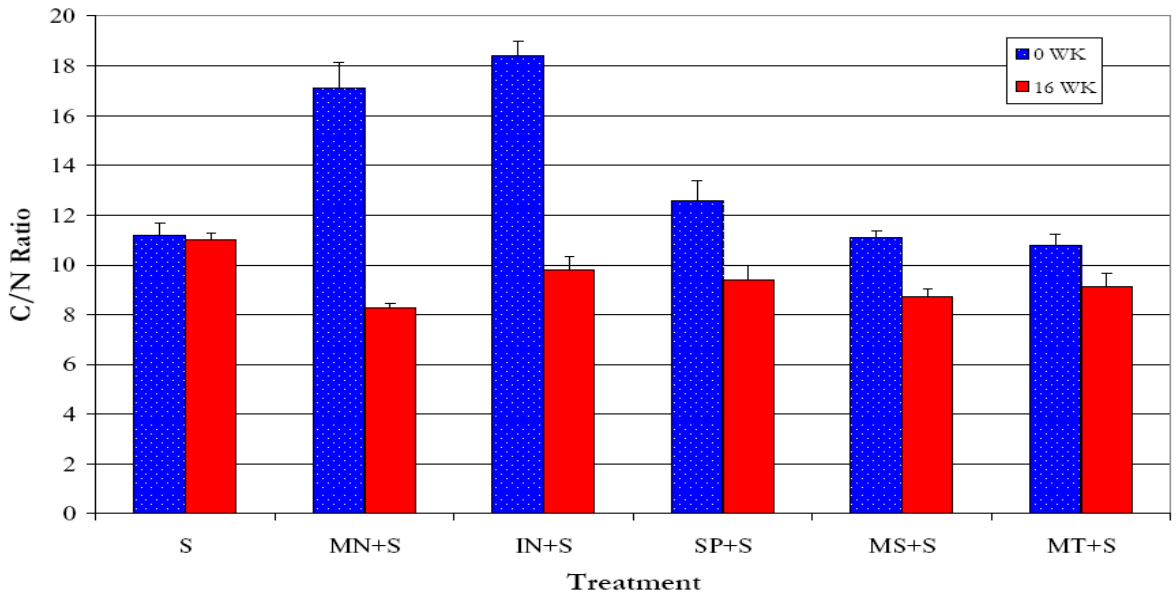


Figure 5.2-8: C:N ratios at the beginning of the experiment, week 0 (blue textured), and at the end of the experiment, week 16 (red). Amendments MN, IN, SP, MS and MT were mixed with soil (S). Error bars are 1 standard deviation.

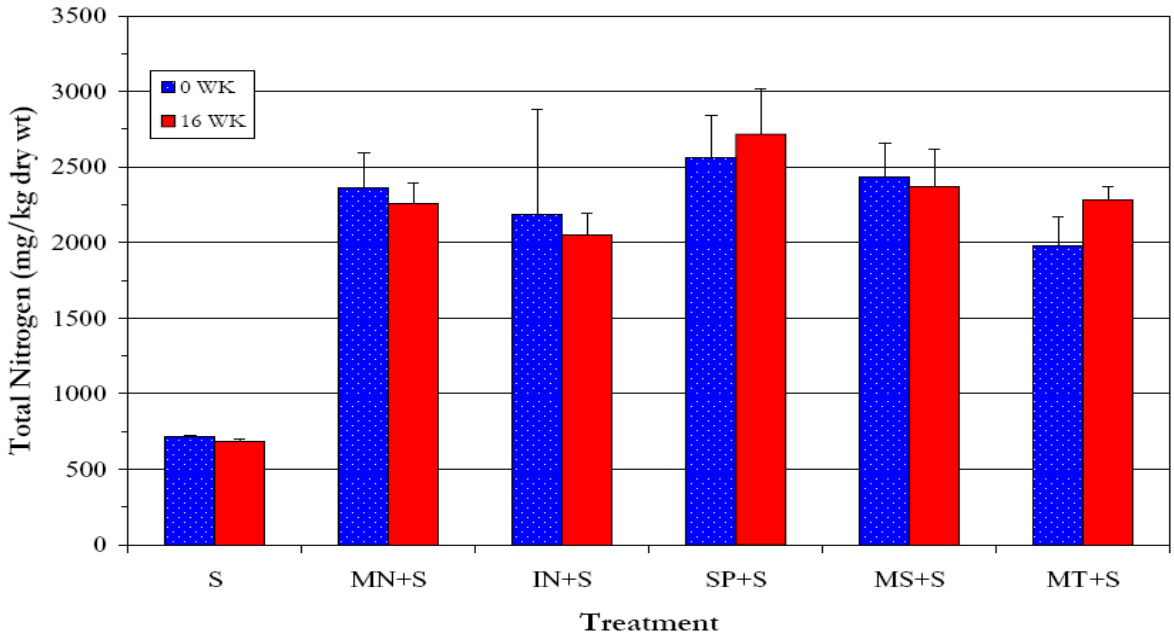


Figure 5.2-9: Total nitrogen concentrations the beginning of the experiment, week 0 (blue textured), and at the end of the experiment, week 16 (red). Amendments MN, IN, SP, MS and MT were mixed with soil (S). Total nitrogen concentrations for amendments include nitrogen concentrations in soil. Error bars are 1 standard deviation.

5.2.2.3 Total Nitrogen

The data for total nitrogen was log transformed to obtain more homogeneous residuals for statistical analysis, although the data presented in Figure 5.2-9 is untransformed. Statistical analysis indicated the main effect, treatment, was statistically significant (Pr_{MAX} = 0.0001). The main effect, time, and the interaction term, trt*time, were not statistically significant (Pr_{MIN} = 0.680). For each treatment, including the control soil, total nitrogen concentrations at the beginning (0 week) and end of the experiment (16 week) were statistically equivalent ((Pr_{MIN} = 0.135). This statistical equivalence indicates a balance between biomass (carbon) losses, which would have created apparent increases in nitrogen concentrations, actual nitrogenous gas losses and nitrogen assimilation.

5.2.3 Discussion

5.2.3.1 Terminology

Nitrogen mineralization is the conversion of organic nitrogen to the inorganic form (total ammoniacal nitrogen and nitrate). Net positive mineralization (NPM) is the terminology used to describe the dynamics of transformations when more inorganic nitrogen has been produced than assimilated, without inferences to specific transformations. Net negative mineralization (NNM) describes the net decrease in inorganic nitrogen concentrations irrespective of transformations (Cambardella et al., 2003). Mechanisms for reductions in inorganic nitrogen concentrations include assimilation (the transformation of total ammoniacal nitrogen into microbial biomass or byproducts), ammonia volatilization of extant total ammoniacal nitrogen, nitrogenous gas losses via nitrification and nitrate reduction processes, leaching losses or plant uptake.

5.2.3.2 Nitrogen Mineralization

Concentrations of net inorganic nitrogen (Figure 5.2-1) for all treatments increased during incubation, although not monotonically. Inorganic nitrogen concentrations decreased from the beginning of the incubation for MN, IN and SP, and reached a minimum during week 4. Figure 5.2-7 (percent inorganic nitrogen mineralized) shows that NNM occurred for up to 12 weeks, for MN, IN and SP, although inorganic nitrogen was still available as a nutrient source, as

indicated in Figure 5.2-1. After 16 weeks, the total amount of inorganic nitrogen mineralized for MN and MT (9-10%) was statistically equivalent ($\alpha = 0.05$). Less nitrogen was mineralized for the other treatments, IN, SP and MS: 4.2, 2.5 and 5.3 percent, respectively. Figures 5.2-3 and 5.2-4, respectively, indicate that there was a deficit of nitrate with respect to the unamended soil during NNM and a continuous decrease in total ammoniacal nitrogen during the whole incubation period for MN, IN and SP. However, for these three amendments, NPM resulted after 16 weeks of incubation. For treatments MS and MT, total ammoniacal nitrogen continually decreased but nitrate concentrations increased for a net positive nitrogen mineralization throughout the incubation.

Nitrate reduction caused the decreases in nitrate since leachate losses and nitrogen uptake by plant roots were not applicable. Denitrification was the most likely mechanism as opposed to dissimilatory nitrate reduction (DNR) as total ammoniacal nitrogen concentrations continually decreased during composting. Ammoniacal nitrogen is the endproduct of DNR, while final product of denitrification is N_2. Assimilatory nitrate reduction also does not appear to have been the predominant mechanism. Although nitrogen assimilation is promoted by excess (labile) carbon, which may have been extant in the MN, IN and SP amended soils (section 5.2.3.5.4), high TAN concentrations inhibit this pathway since reducing nitrate for biosynthesis requires more energy than assimilating extant ammoniacal nitrogen (Chapter 2.7.2). Figures 5.2-3 & 5.2-6 indicate elevated TAN levels.

The decrease in total ammoniacal nitrogen can be attributed to primarily nitrogen assimilation, with ammonia volatilization being a contributing factor. The initial concentrations (week 0) were related to the total ammoniacal nitrogen loading rates (Table 4.2-4).

Denitrification is promoted by low oxygen and low pH conditions; ammonia loss is enhanced by high pH and low oxygen environments. Equations 5-1 (denitrification) and 5-2 (ammonia formation) are the general chemical equations that govern these reactions. Equation 5-1 shows the overall stoichiometry of a microbially mediated, multi-step process; equation 5-2 shows a reversible chemical process.

$$2NO_3^- + 5H_2 + 2H_3O^+ \rightarrow N_2 + 8H_2O \qquad (5\text{-}1)$$

$$NH_3 + H_2O \leftrightarrow NH_4^+ + OH^- \quad (5\text{-}2)$$

Low oxygen levels, induced by high microbial activity in the amended soils (inferred by the reduction in total ammoniacal nitrogen concentrations and net negative nitrate levels), appear to have been the cause of NNM in the MN, IN, and SP amended soils. If low oxygen conditions did not evolve, increases in nitrate concentrations would have been exhibited along with decreases in TAN. Less composted OM tends to contain higher amounts of easily decomposable carbon and nitrogen moieties (such as carbohydrates, sugars and proteins) resulting in higher microbial activity when mixed with soil (Zaccheo et al., 1993; Bernal et al., 1998a & c), resulting in hypoxic conditions (Miller et al., 1985; Katayama et al., 1987). As aerobic decomposition progresses, the more labile fraction is consumed, leaving the more recalcitrant nutrient fraction. This is reflected in the shift to lower decomposition rates as OM degradation (eg, composting) progresses (Chèneby et al., 1994; Bernal et al., 1998a & c; Benito et al., 2005; Gale et al., 2006).

Additionally, although nitrogenous gas volatilization was not monitored during the incubation, ammonia volatilization may have contributed to nitrogen loss in the MN, IN and SP amended soils. Alkaline conditions favor ammonia formation (eq. 5-2). The pH values of the amendments (Figure 5.1-19) were alkaline (about 7.5 to 8.8) and the pH of field soil samples (Figure 5.3-2) was almost neutral (about 6.6). Consequently, as the amendment/soil mixes in the incubation study were probably, at most, only slightly alkaline, and the pk_a for ammonia is 9.8, the thermodynamic barrier for elevated ammonia volatilization would have been significant. However, the initial high concentrations of TAN would have created a large chemical gradient between the soil solution and atmosphere promoting ammonia volatilization. Ammonia volatilization can occur in neutral or slightly acidic soils in the presence of high concentrations of TAN (du Plessis et al., 1964) or rapidly decomposing OM (Davet, 2004). Conversely, the organic and inorganic fractions of soil adsorb ammonium, although the efficacy is a function of mixing efficiency and mixing depth (in the field) (Thompson et al., 2002).

Only soil total ammoniacal nitrogen and nitrate/nitrite levels were determined in the incubation study. Losses due to leaching were not applicable since the incubations were

conducted in plastic (polyethylene) bags, and nitrogen losses due to uptake by plants were not applicable since no plants were grown in the soil. Since contributions of gas loss via denitrification and ammonia volatilization were unknown, the mineralization rates that were determined may have been underestimated. The calculated percent nitrogen mineralization values in the present study, except for MT, were lower than those projected for the amendments; for MT, the measured and hypothesized values were similar (Table 5.2-1). Additionally, increasing duration of composting did not reduce the mineralization rates as other investigators have observed (Zaccheo et al., 1993; Robertson et al., 1995; Hartz et al., 2000). Explanations for the results of the mineralization study are discussed below.

Table 5.2-1: Percent organic nitrogen mineralization based on initial organic nitrogen content: projected and measured

Amendment	Projected (%)	Measured (%)
Manure	30	9
Compost IN	25	4
Compost SP	20	2.5
Compost MS	15	5.5
Compost MT	8	10

5.2.3.3 Manure

In the present study, the manure solids were a mixture of fresh and aerobically decomposed manure. When estimating percent nitrogen mineralization of manure for the present study, incubation experiments involving fresh or aerobically treated swine manures were considered (Appendix C). Castellanos et al. (1981) incubated swine manure amended soils for 10 weeks at 23^{O} C, and reported about 34% nitrogen mineralization with no indications of NNM. Chae et al. (1986) reported nitrogen mineralizations of 31% and 35% for swine manures in Lester and Webster series soils, respectively, after 16 weeks incubation at 30^{O} C; NNM occurred for the first 2 weeks in the Webster soil. (Insufficient data was presented to determine whether NNM occurred within the first 4 weeks for the Lester soil. After 4 weeks, NPM occurred.)

Using the data from the study of Bernal et al. (1992), the present author calculated a

percent nitrogen mineralization of approximately 5% after 9 weeks of incubation at 25^{O} C, which was the average for fresh and aerobically treated swine manures. From these investigations 30% mineralization was hypothesized for the manure (MN) in the present investigation. However, 9% mineralization was measured, after 16 weeks at 23^{O} C; NNM occurred for the first 4 weeks, with a spike at 12 weeks (Figure 5.2-2).

In the aforementioned investigations, nitrogen mineralization was not necessarily calculated in the same manner. In all of the studies, except Chae et al. (1986), nitrogen mineralization calculations included subtracting initial concentrations of inorganic nitrogen from total inorganic nitrogen. Chae et al. (1986) used a leaching technique to determine the concentration of soluble inorganic nitrogen, although at the end of the study a soil analysis was also included to provide a comprehensive inorganic nitrogen accumulation for the whole incubation study, which was 26 weeks. Consequently, percent mineralizations for less than the whole incubation period, calculated by the present author, were probably underestimates, as only the water soluble inorganic nitrogen was included.

Incubation conditions and the composition of the manure affect mineralization rates, the latter being governed by the feed characteristics (Sørensen et al., 2003; Powell et al., 2006) and the state/treatment of the manure prior to the incubation. Higher incubation temperatures in the study of Chae et al. (1986) may have contributed to elevated mineralization rates, since nitrogen mineralization rates increase with temperature (Terry et al., 1981; Tachimoto, 1995; Agehara et al., 2005) due to increased microbial activity (Avrahami et al, 2003; Bekku et al., 2003). Chae et al. (1986) used an incubation temperature of 30^{O} C, which was at least 5^{O} C higher than in the other referenced investigations, including the present study.

With respect to compositional characteristics, Parnaudeau et al. (2004) noted that for municipal wastewater sludges, those with high C:N ratios (maximum 18.8) and a very low lignin-like fraction caused high carbon mineralization and NNM in the early stages of decomposition. In the present study, the C:N ratio of MN was 20.4, and the manure induced NNM primarily during the early stages of the incubation. Therefore, the manure may have contained a low lignin-like fraction, which is consistent with the relatively low lignin fraction in swine manures reported by others (Hilliard et al., 1978/1979; Ngian et al., 1978/1979; Richard, 1996).

Castellanos et al. (1981) did not provide a description of the manure in their study. Chae

et al. (1986) described their swine manure as being "fresh". Bernal et al. (1992) incubated fresh swine manure and manure that had been aerobically incubated for 6 months prior to the mineralization study. As previously described, the manure solids in the present study, were a mixture of fresh and aerobically treated manures.

In the present study, the mineralization of the manure (MN) at 9 weeks was approximately 4% (Figure 5.2-7), which is similar to the results of Bernal et al. (1992), although the temporal changes were not necessarily similar. In the present study, Figure 5.2-6 shows a sharp decline in total ammoniacal nitrogen and Figure 5.2-5 shows the initial reduction of nitrate followed by nitrification. NNM was dominant in the early stages of the study, whereas NPM was predominant in the latter phases. The temporal trends for total ammoniacal nitrogen and nitrate were similar to those of Bernal et al. (1992). However, the aerobically treated manure exhibited only NPM and the fresh manure induced NNM for most of the incubation.

While many disparate organic fractions have been reported to affect mineralization dynamics (Rowell et al., 2001; Parnaudeau et al., 2004; Jensen et al., 2005), soil characteristics also impact the processes (Chae et al., 1986; Stotzky, 1986; Hébert et al., 1991). The reported effects of carbonates of calcium and magnesium on nitrogen mineralization have been variable. Stotzky (1986) concluded that mineralization enhancement does occur due to the buffering capacity of the carbonates. However, Hartz et al., (2000) amended a calcareous and non-calcareous soil with either poultry manure or municipal waste compost and reported that the nitrogen mineralization rates in the calcareous soil were slightly lower than in the non-calcareous soil. Given the variability in the literature, it is unclear what effects the calcium carbonate (extant in the soil and added as lime) may have had on nitrogen mineralization rates in the present investigation.

Carbon and nitrogen mineralization are affected by soil texture, albeit inconsistently (Tester et al., 1977; Stotzky, 1986). Type of clay also has an effect on mineralization. Nitrification is enhanced by montmorillonite via its buffering capacity and by the clay's ability to increase microbial survival, whereas illite and vermiculite may depress nitrification by their ability to fix ammonium (Tester et al., 1977; Stotzky, 1986). In the present study, the soil was kaolinitic. Compared to higher CEC clays, the relatively low CEC of kaolin would have a low capacity to enhance buffering capacity or microbial survival; kaolinite does not fix ammonium.

Castellanos et al. (1981) used a calcareous, montmorillonitic soil, which would have tended to enhance nitrification due to the buffering capacity of the soil, although an alkaline pH (inferred by the calcareous soil) would have increased ammonia volatilization. Bernal et al. (1992) mixed manures with calcareous, illitic soil. The lower apparent mineralization rates in their study may have been due to ammonium fixation by illite (ammonium interlayer fixation by illite/vermiculite is not accounted for when determining inorganic nitrogen by neutral potassium salt saturation – the procedure used by Bernal et al., 1992.) and alkaline soil (pH ~7.4), which would have promoted ammonia volatilization. Chae et al. (1986) used five different soils, and although the soils selected for comparisons (Lester and Webster series) were the most similar to the soil used in the present study (Appling series), the other soils had certain characteristics that were similar to soils used in the other referenced investigations (such as being calcareous or montmorillonitic). For all five soils (Chae et al., 1986) the range for nitrogen mineralization was approximately 12.5 – 43%, with an average of 32%, for a 16 week incubation.

5.2.3.4 Manure Plus Bulking Agents

The addition of a higher C:N organic material (such as wood chips and wheat straw) to animal wastes with a lower C:N ratio tends to decrease nitrogen mineralization rates, compared to the wastes alone although the differences may not be statistically significant (Mahimairaja et al., 1994; Chantigny et al., 2001). The trends reported by Chantigny et al. (2001) were for amendments mixed with soil, while Mahimairaja et al. (1994) incubated poultry manure/bulking agents without soil. In the present study, the addition of the chipped wood/leaf bulking agent to the swine waste solids (IN) produced lower nitrogen mineralization than MN over 16 weeks. The amount of mineralized nitrogen declined from about 9% to 4% (Figure 5.2-7), and the inorganic nitrogen concentration (Figure 5.2-4) decreased from about 350 to 165 mg/kg dry wt.

Possible contributors to the reduction in nitrogen mineralization are the particle size, C:N ratio and the proportion of recalcitrant OM, all of which affect microbial activity. In the present study, the particle sizes of the wood chips and leaves were much larger than that of the manure. The greater volume:surface area ratio of the bulking agent, especially the wood chips would have tended to decrease decomposition rates.

Nitrogen mineralization/immobilization dynamics are regulated by carbon and nitrogen

availability. The C:N ratio has traditionally been used as an index for decomposition dynamics, although it is an inconsistent indicator (Cambardella et al., 2003; Jensen et al., 2005) The demarcation between the predominance of nitrogen immobilization and mineralization is not a specific C:N ratio, but actually a wide range (see Chapter 2.2.4.1), and is dependent upon the characteristics of the OM. In the present study, the addition of the chipped wood/leaf bulking agent increased the C:N ratio of the swine waste solids (MN) from 20.4 to 27.5 (initial mix – IN), and both amendments induced net NPM after 16 week, although the mineralization rates oscillated between positive and negative over that time period (Figure 5.2-7).

Augmenting recalcitrant fractions (eg, lignin) in the manure, is another possible cause of reduced nitrogen mineralization. Lignin, one of the regulating variables in decomposition dynamics (Melillo et al., 1982; Mafongoya et al., 2000; Rowell et al., 2001; Sariyildiz et al., 2003; Parnaudeau et al., 2004), is a three dimensional polymer of phenyl-propane units that is not readily biodegradable compared to other organic components (Wilson, 1985; Adl, 2003). Although the composition of manures vary with feed characteristics (Sørensen et al., 2003; Powell et al., 2006), and the lignin level of wood/leaves can vary significantly, in general, swine manure contains less lignin than wood or leaves: manure contains about 2-6% (Hilliard et al., 1978/1979; Ngian et al., 1978/1979; Richard, 1996), wood approximately 18-40% (Francis et al., 1980; Adl, 2003) and freshly fallen leaves about 10-39% (Melillo et al., 1982; Sariyildiz et al., 2003).

5.2.3.5 Compost

5.2.3.5.1 Overview

In the present study, the trend of increasing nitrogen mineralization with composting time did not confirm other studies indicating decreasing mineralization (Castellanos et al., 1981; Tyson et al., 1993; Robertson et al., 1995), although it is similar to the results of Bernal et al. (1998a). Gale et al. (2006) reported that composting does not necessarily decrease nitrogen mineralization and that it is feedstock dependent: decreasing with rabbit manure and yard trimmings, but increasing with dairy solids. While decreasing carbon mineralization rates with increasing composting time have also been observed (Bernal et al., 1998a & c; Benito et al., 2005), the relationship between carbon and nitrogen mineralization is inconsistent. Positive and

negative correlations have been reported (Robertson et al., 1995; Bernal et al., 1998a), as well as lack of correlations (Cambardella et al., 2003; Parnaudeau et al., 2004). Both rates tend to decrease as nutrient availability diminishes, although not necessarily monotonically (Cambardella et al., 2003).

In the present study, composts MS and MT were composted for the longest times and also went through a full thermophilic phase and therefore would have had less available nutrients than MN, IN, and SP. Treatments MS and MT would thus have stimulated microbial activity less within a short period than the other treatments and tended not to create low oxygen conditions. Manure-based amendments with readily available carbon promote denitrification due to higher microbial activity (which creates low oxygen conditions), whereas a slower release of carbon favors nitrogen immobilization and minimizes denitrification (Hadas et al., 1994a; Calderón et al., 2005). Reductions of nitrate (Figure 5.2-5), along with decreases in total ammoniacal nitrogen (Figure 5.2-6), are apparent for MN, IN, and SP, indicating higher microbial activity. Figure 5.2-5 infers denitrification and Figure 5.2-6 implies nitrogen assimilation. Composts MS and MT showed a decrease in total ammoniacal nitrogen levels, but a consistent increase in nitrate concentrations, indicating a lower microbial activity level, with the total ammoniacal nitrogen being nitrified and/or assimilated.

5.2.3.5.2 Similar Investigations

The investigations of Robertson et al. (1995), Bernal et al. (1998a) and Cambardella et al. (2003) will be discussed because of dissimilar results, and will be compared with the present experiment since in all four studies, compost was sampled at several points during the process, instead of just an initial and final sampling as in most other investigations. In the study of Robertson et al. (1995), fowl manure, grass clipping and coal were co-composted for up to 16 weeks. Mixtures composted for 0, 2, 4, 8, 12 and 16 weeks were incubated in soil for 9 weeks. The data indicated decreases in carbon mineralization and inorganic nitrogen concentrations with increasing composting time. However, maximum inorganic nitrogen levels were produced for the 2 week compost. The nitrogen data was re-calculated as percent mineralized, by this author (Appendix B).

Cambardella et al. (2003) composted swine waste/corn stover feedstocks for 0, 2, 4 and

6 weeks, and incubated them for 28 days. In one experiment (Trial II), feedstocks with initial C:N ratios (compost+soil) of 15.3 (A) and 22.6 (B), the C:N ratios being adjusted with corn stover, induced NNM throughout the incubation for every compost batch (0, 2, 4, and 6 weeks), except for compost A at week 0 when slight NPM was induced. The relationship between time of composting and nitrogen mineralization was inconsistent for A and B, although the 4 week composts induced the least NNM for both. Additionally, carbon mineralization generally decreased with composting time.

Bernal et al. (1998a) co-composted wastewater residuals and cotton wastes for approximately 16 weeks. Composts collected at three sampling points were aerobically incubated for 10 weeks. The composts labeled I, E and M were similar to IN, MS and MT, respectively, in the present study. Compost I was the initial undecomposed mix and equivalent to IN; M, "aged" two months after pile temperatures had decreased to ambient (49 days), was similar to MT. Compost E was defined as when the pile temperature returned to ambient, which was somewhere between MS (returned to mesophilic temperature, 45^{O} C) and MT, but closer to MS. The authors reported decreasing NNM with composting time, which is similar to the results in the present study (Figure 5.2-7). The trends of decreasing total ammoniacal nitrogen concentrations and increasing nitrate levels were also common to both studies. Although the nitrogen mineralization of M (9.1%) was greater than E (4.8%), the authors did not indicate that there were differences, only that they were similar. It is apparently a matter of interpretation as to whether the differences were "meaningful" as there were no statistical comparisons.

Compost I induced NNM for most of the 10 week incubation, while IN caused NNM through 10 weeks of the 16 week incubation. Except for the initial 2 day period for E, composts E and M induced NPM the entire incubation, which is also similar to the results in the present study (Figure 5.2-7). This infers that the microbial activity (reflected by the carbon mineralization rate) for I was significantly greater than either E or M, which were similar, and would explain the induction of NNM by I.

Bernal et al. (1998a) used the Rutgers static pile method for composting (Finstein et al., 1992), with the objective of limiting the maximum temperature to 55^{O} C. Temperature/time profiles were not provided by Robertson et al. (1995) or Cambardella et al. (2003). The protocol of Robertson et al. (1995) involved mixing the compost piles at every sampling point (0, 2, 4, 8,

12 and 16 weeks). Cambardella et al. (2003) used insulated bioreactors for composting.

5.2.3.5.3 Feedstock

Disparate concentrations of labile carbon and nitrogen in the initial compost mixtures may help explain the difference in results of Robertson et al. (1995), Bernal et al. (1998a), Cambardella et al. (2003) and the present study. The study of Bernal et al. (1998a) and the present study were similar in that the initial composts induced NNM that transformed into NPM as the duration of composting increased. In the study of Robertson et al (1995), NNM did not occur. NPM was induced with the initial mixtures, but subsequently decreased with increasing duration of composting. In the study of Cambardella et al. (2003), NNM dominated throughout most of the incubation.

The NNM that occurred with the less composted feedstocks in the present study and of Bernal et al. (1998a) indicates that a large pool of labile carbon was initially extant. In the present study, the manure likely contained high level of available carbon (as discussed previously). In the study of Bernal et al. (1998a), the decrease in carbon mineralization with increasing composting duration, which was opposite that of the nitrogen mineralization trend, appears to confirm this speculation. Initial concentrations of water soluble carbon are highly correlated with carbon mineralization rates (Bernal et al., 1998b). Large pools of available carbon in the initial mixtures would have caused high microbial activity, creating low redox conditions and consequently assimilation and denitrification (Calderón et al., 2005). In the present study, the trends of decreasing total ammoniacal nitrogen and net negative amounts of nitrate confirm this assumption. With the manure and less composted feedstocks, as composting time increased, the pool of available carbon diminished, reducing microbial activity and thus oxygen deficits. Consequently, NNM was transformed to NPM. Conversely, the fact that no NNM occurred in the study of Robertson et al. (1995) indicates that a smaller pool of labile carbon was initially present, which was insufficient to cause the high microbial activity necessary to induce low redox conditions. Manure-based amendments with readily available carbon promote denitrification due to higher microbial activity (creating low oxygen conditions), whereas a slower release of carbon favors nitrogen immobilization and minimizes denitrification (Hadas et al., 1994a; Calderón et al., 2005). Thus, the trend of increasing or decreasing nitrogen mineralization may depend upon the size of the initial pool of labile carbon.

In the study of Cambardella et al. (2003), NNM predominated throughout the incubation period, but the authors reported an inconsistent relationship between composting time and nitrogen mineralization. This inconsistency has several contributing factors. The initial NNM (and initial, low NPM for feedstock A) may have been due to the presence of a large pool of available carbon inducing high microbial activity. Additionally, the duration of incubation affects apparent nitrogen mineralization rates. In the present study, Figure 5.2-7 shows that for composts IN (0 weeks) and SP (19 day), NNM lasted for many weeks prior to NPM, whereas Cambardella et al. (2003) used an incubation period of 28 days. Therefore, it is possible that a longer incubation time might have exposed a transition from NNM to NPM. Another contributing factor to the inconsistency may have been the duration of composting. If the compost periods sampled (0, 2, 4 and 6 weeks) were primarily within the more dynamic period of decomposition, more transient nutrient availability conditions might have prevailed.

Although nutrient availability decreases as composting progresses, declines in soluble carbon during composting are not necessarily monotonic (Sánchez–Monedero et al., 1999; Hsu et al., 2001), nor is nitrogen (García-Gómez et al., 2005), as catabolic processes release nutrients from OM. The former phenomenon may explain the longer NNM induced by SP in the present study, compared to the initial mix (IN): a temporary spike in soluble carbon could have induced a short term peak in microbial activity causing increased nitrogen assimilation and denitrification. In the study of Cambardella et al. (2003), the minima in NNM induced by the 4-week composts, may have been due to a release of labile nitrogen. The release of nitrogen could also explain peak nitrogen mineralization for the 2-week compost in the study of Robertson et al. (1995).

5.2.3.5.4 C:N Ratios

Carbon to nitrogen ratios are used as indicators of decomposition dynamics with high ratios suggesting NNM and low values indicating NPM, due to nitrogen being limited in the former and in excess in the latter cases. Decreases in the rate of reduction of C:N ratios also signify reductions in nutrients or nutrient availability. However, low oxygen conditions can affect the C:N threshold demarcating net microbial nitrogen assimilation and mineralization and affect NNM and NPM. Oxygen deficits promote nitrogenous gas losses which may reduce apparent mineralization. Additionally, microbial yield coefficients are reduced in low oxygen

environments since less energy per unit substrate is produced resulting in the requirement for greater amounts of energy sources (ie, carbon). Consequently, the C:N ratio in aerobic conditions, which may be indicative of nitrogen assimilation/mineralization may not be applicable in low oxygen conditions.

In the experiment of Cambardella et al. (2003), C:N declined from 15.3 to 10.1 and from 22.6 to 13.1 for two different composts (A & B), and NNM predominated throughout most of the incubation period. The C:N changed from 20.8 to 16.9 in the study of Robertson et al. (1995), and only NPM was indicated. In the study of Bernal et al. (1998a)., the initial C:N ratios of I, E and M were 21.0, 9.9 and 9.4, respectively. The precipitous drop from a high ratio to a low ratio caused a concomitant NNM to NPM transformation. These trends in the present study are similar to those of Bernal et al. (1998a).

In the present study, the C:N ratio (Figure 5.1-5) decreased as composting time increased (27.6, 17.3, 12.3 and 11.0) for IN, SP, MS and MT, respectively) with an associated non-monotonic increase in nitrogen mineralization (Figure 5.2-2), and a transition to NPM throughout the incubation from primarily NNM when the C:N ratio decreased from 17.3 (SP) to 12.3 (MS). Similarly, MN with a C:N ratio of 20.4, exhibited an NNM to NPM transition during the incubation. Therefore, the demarcation between nitrogen mineralization and assimilation may have been between 17.3 and 12.3. The associated amendment/soil C:N ratios were 12.6 and 11.1 (Figure 5.2-8). Above the threshold, assimilation would have predominated and below the threshold mineralization would have dominated. As the final amendment/soil C:N ratios of MN, IN and SP were less than 11.1, the transition from NNM to NPM may have been partly due to an assimilation-to-mineralization transition.

5.2.3.6 Amendment:Soil Ratio

In addition to the compositional differences that may have contributed to disparate nitrogen mineralization dynamics, the amendment:soil (Am:S) ratios may have also have been a factor. Soil microbial activity generally increases with increasing application rates of OM/nutrients (Tester et al., 1977; Nishio et al., 1980; Saviozzi et al., 1993; Pascual et al., 1998), and high application rates can lower redox potentials (Miller et al., 1985). Hébert et al. (1991) observed that in a sandy soil at low application rates, the less composted OM induced a higher

mineralization rate than the more composted OM. However, at a high loading rate, the reverse occurred with the less composted OM inducing NNM apparently due to a higher microbial activity.

In the present study, the Am:S rate was higher than the field recommended rate due to the inhomogeneity of the composts (Chapter 4.4); the field rates were based on projected nitrogen mineralization values. The high Am:S ratio, resulting in high nutrient loading, may have contributed to the initial NNM of MN, IN and SP treatments and the reduced rate of mineralization.

However, although the Am:S ratio (Appendix D) was higher than the application rates of the amendments in the field study, the nitrogen concentration (1.7 mg total N/g soil dry wt) was not unusually high for incubation studies. Other investigators have used nitrogen loading rates of up to 1.6 to 2 mg total N/g soil dry wt (Hadas et al., 1996; Cambardella et al., 2003).

5.2.3.7 Moisture Content

In the present study, an initial condition was to create moisture levels in the soil/amendment mixture equivalent to 95% container capacity in the soil and about 50% moisture in the amendment. This was a theoretical calculation to determine total water contents of the mixtures, and subsequently amounts of water that might be needed to be added to mixtures to achieve the target levels. This produced total moisture contents of varying levels (Figure 5.2-10) in the mixtures since different amounts of amendment were added to the soil. The total water contents of soil/amendment mixtures amended with IN, SP, MS and MT were similar (95% container capacity of soil and 50% moisture content of amendment). The MN amended soil contained higher water content (Appendix D).

High moisture contents can contribute to lower mineralization rates by inducing emissions of nitrogenous gas (N_2, N_xO_y, NH_3). Nitrogen mineralization increases as (soil) moisture content increases up to approximately field capacity (Gonçalves et al., 1994; Agehara et al., 2005), the specific demarcation (-0.03 to –0.01 MPa) being dependent upon soil characteristics and nutrient levels (Myers et al., 1982). However, as moisture levels increase, at high moisture conditions, the availability of oxygen for microbial respiration decreases. Eventually the environment becomes anoxic and nitrogenous moieties become the source for

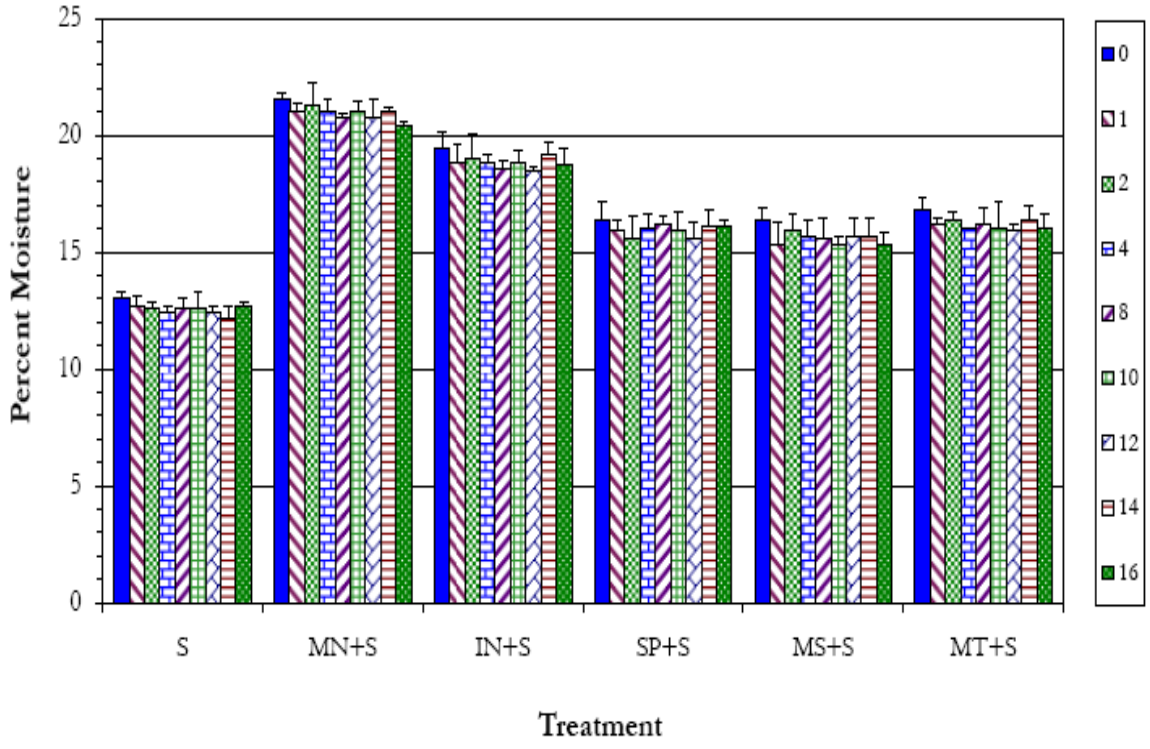


Figure 5.2-10: Moisture contents of soil (S) and amendment (MN, IN, SP, MS, MT)/soil mixtures. Time legend is in weeks. Error bars are 1 standard deviation.

electron acceptors and oxides of nitrogen are reduced, producing nitrogenous gases as intermediate or final products. Additionally, as low oxygen conditions inhibit nitrification, ammonia emissions are promoted.

Fischer et al. (2005) measured denitrification in soils amended with liquid lagoonal swine wastes that created moisture levels up to and exceeding container capacity. The authors reported that denitrification became detectable at a moisture condition of 96% container capacity (the previous lower analytical point was 77% container capacity), and significantly increased at a moisture level exceeding container capacity. Similarly, Maag et al. (1999) reported that in soils amended with either cattle or swine waste slurries, denitrification was relatively low up to 100% soil field capacity, but sharply increased when field capacity was exceeded.

Ammonia formation is also enhanced at higher moisture contents. Cabrera et al. (1994)

reported that ammonia losses from poultry litter continually increased as the water content increased from less than wilting point (WHC) to almost field capacity (WHC), although the increases were not necessarily statistically significant. Conversely, Sharma et al. (1989) reported that ammonia losses at 75% and 100% field capacity were less than at 50% field capacity for nitrogen sources mixed with soil. The authors speculated that ammonia volatilization was reduced at higher moisture levels because the water facilitated the dispersion and subsequent adsorption of ammonium onto soil particles. An additional explanation may be that the water also acted as a sink for the ammonia, as ammonia is soluble in water.

Figure 5.2-7 shows that NNM occurred for MN, IN and SP. Figure 5.2-5 indicates that denitrification occurred in the three amended soils with the trends being similar, although the moisture content of MN was greater than IN and SP. Figure 5.2-6 indicates sharp decreases in TAN concentrations following the initial sampling event for MN, IN and SP, which were due to nitrogen assimilation and possibly ammonia volatilization. MN amended soil, with the highest moisture content, produced the greatest reduction in TAN. However, although high moisture content may have contributed to nitrogenous gas losses (lowering apparent nitrogen mineralization rates), it does not appear to have been the major contributing factor. The moisture contents of IN, SP, MS and MT were similar, although the temporal trends for nitrogen mineralization were different. Additionally, the control soil with a moisture level of 95% container capacity did not appear to induce nitrogenous gas losses to a significant extent as indicated by 1) the lack of decrease in inorganic nitrogen over time (Figure 5.2-1), 2) the absence of nitrate decrease over time (Figure 5.2-2), which would indicate nitrate reduction and 3) the constant levels of total ammoniacal nitrogen (Figure 5.2-3), indicating low ammonia volatilization. NNM appeared in only the earlier stages of the incubation in MN, IN and SP amended soils. As the moisture contents of the amendment/soil mixtures remained fairly consistent throughout the study (Figure 5.2-10), no transformations to NPM should have occurred if the low oxygen conditions were caused by high moisture contents. Therefore, high microbial activity (ie, aerobic respiration) due to high nutrient content was the cause of the NNM, resulting in lower nitrogen mineralization rates. However, higher moisture contents may have lowered the NNM threshold by reducing oxygen diffusion rates.

5.2.3.8 Laboratory Versus Field

In the present study, the laboratory incubation conditions did not attempt to mimic field conditions, and therefore, the nitrogen mineralization dynamics may not have been accurate predictors for field applications. The laboratory conditions did not account for plant growth, simulate fluctuations in temperature or precipitation, or allow for movement of nitrogenous species down into the soil profile as in field situations. Johnson et al. (1980) attributed the disparate results of net nitrogen mineralization in a laboratory study and immobilization in a field study to the presence of plants in the field study. Debosz et al. (2002) reported that over the short term (weeks) laboratory and field incubations produced similar mineralization rates., however, divergence between the inorganic nitrogen levels increased the longer the incubations continued. Whereas the laboratory study showed increasing soil inorganic nitrogen, the field study showed non-monotonic decreases. The authors concluded that the latter was due to plant uptake of nitrogen and higher ambient temperatures (compared to laboratory incubation temperature) that would have stimulated microbial activity. They also speculated that cycles of wetting and drying, which would have occurred in the field setting, might have increased microbial activity (Balesdent et al., 2000). Leaching of nitrate could also have contributed to lower inorganic nitrogen concentrations.

Due to the differences between laboratory and field conditions, plus the higher Am:S rates in the laboratory experiment, it's uncertain how applicable the laboratory mineralization rates were to field conditions. Laboratory results may reflect only the potential and not actual carbon and nitrogen mineralization dynamics under field conditions (Chèneby et al., 1994).

5.2.3.9 Calorific Values

The calorific values for the amendments did not predict relative mineralization rates in the incubation study, as might have been inferred from the original hypothesis that calorific values would be indicative of the potential for enhanced plant response or soil properties. While the calorific values for SP and MS on the day of field application were high (Figure 5.1-21 & 5.1-22), the concentration of inorganic nitrogen (Figure 5.2-1) and mineralization rates (Figure 5.2-2) were low. Conversely, MN and MT induced the greatest amount of inorganic nitrogen, although the calorific values on the day of application were low (Figure 5.1-21) or inconsistent (Figure 5.1-22) with the calorific trends.

5.2.4 Summary of Conclusions

The estimated nitrogen mineralization rates predicted for the amendments did not correspond to the results of the incubation study, with the exception of MT. Amendment and soil characteristics, incubation conditions, Am:S and moisture levels affect mineralization rates. Most investigators, probably due to practical reasons, also do not characterize the wastes and soil in detail. Consequently, cogent comparisons between investigations are difficult, and thus predicting mineralization rates based on other investigations is challenging.

5.3 Field Study - Soil Properties

5.3.1 Chemical

5.3.1.1 Electrical Conductivity

The electrical conductivity (EC) of the soil plots (Figure 5.3-1) decreased over time. The values measured (maximum ~0.2 dS/m) would not induce deleterious plant response since a level of about 5 dS/m has been reported to be the threshold for sorghum (Bresler et al., 1982).

In the present study, the trend for the initially high values of the organically amended soil plots generally corresponded to the hierarchy of EC values for the amendments (Figure 5.1-20). The initially high EC for the plot amended with mineral fertilizer was probably due to the high input of cations and anions associated with the fertilizer (TAN, nitrate and potassium). However, it appears that the inorganic and organic amendments were only partially responsible for the initially high values of EC, although EC values greater than the negative control (F, SP, MS and MT) were due to the amendments. After about 6 weeks all of the plots, except MT, appeared to achieve equilibrium, and the EC levels were similar, including the negative control. MT maintained equilibrium after approximately 10 weeks.

The initially high levels for all of the plots appear to have been perturbations of a lower soil EC equilibrium. The fact that the NF plot, as well as the amended plots, varied relatively little once the lower equilibrium had been (re)established indicates that the field preparation/climatic conditions were responsible for the high initial EC values. Additionally, while the lower equilibrium achieved in the negative control was very stable over the remaining sampling periods, the EC of the organically amended plots fluctuated more, indicating that the latter plots may have been more susceptible to environmental fluctuations due to the higher potential for microbial activity.

Soil tillage, which occurred on the day of sowing, exposed more soil to potential physical erosion (Brady, 1990) and also increased soil microbial activity (Balesdent et al., 2000). Increased physical erosion (wind and precipitation) may have enhanced dissolution of inorganic particles, while increased microbial activity would have increased the decomposition of OM, releasing organic acids and inorganic ions (Manios et al., 1989; Inbar et al., 1993). Plant uptake and precipitation/irrigation, which would have enhanced leaching into the soil profile, would have reduced the temporary spike in EC to pre-tillage levels. Organic contributors may also have volatilized or have been further metabolized contributing to the reduced EC.

5.3.1.2 pH

Figure 5.3-2 shows the pH of the soil plots. The very high within-treatment variability precluded any definitive conclusions except that it appears that over the course of the year, the pH decreased. Given that the negative control (NF) reacted in the same manner, it seems that this trend was regulated by environmental factors rather than amendment characteristics (see Chapter 5.5.3.1).

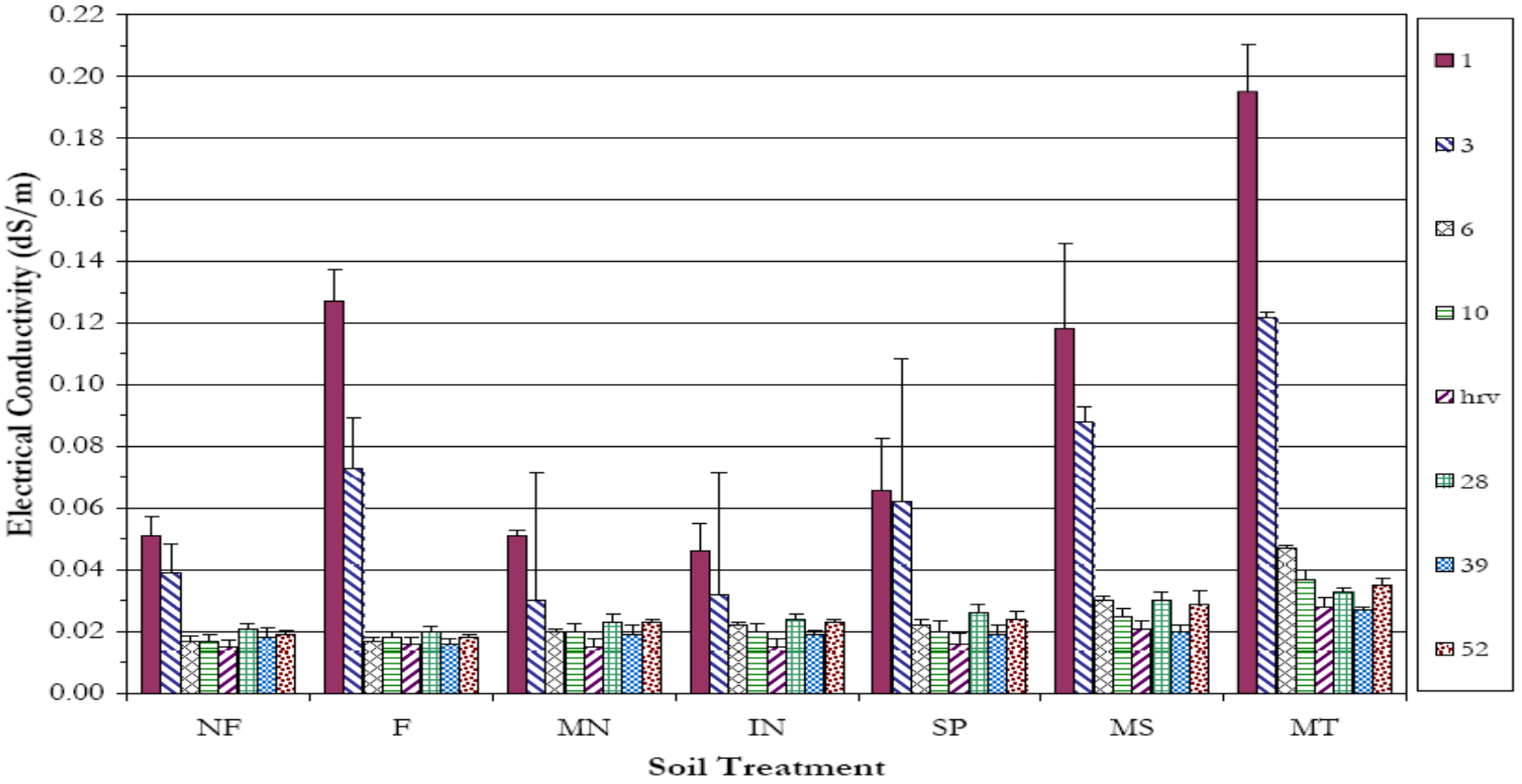


Figure 5.3-1: Electrical conductivity of soil measured over 12 month period following planting for each soil treatment: NF (unfertilized), F (fertilized), MN (manure), IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day). Time legend is in weeks; hrv=harvest. Error bars are 1 standard deviation.

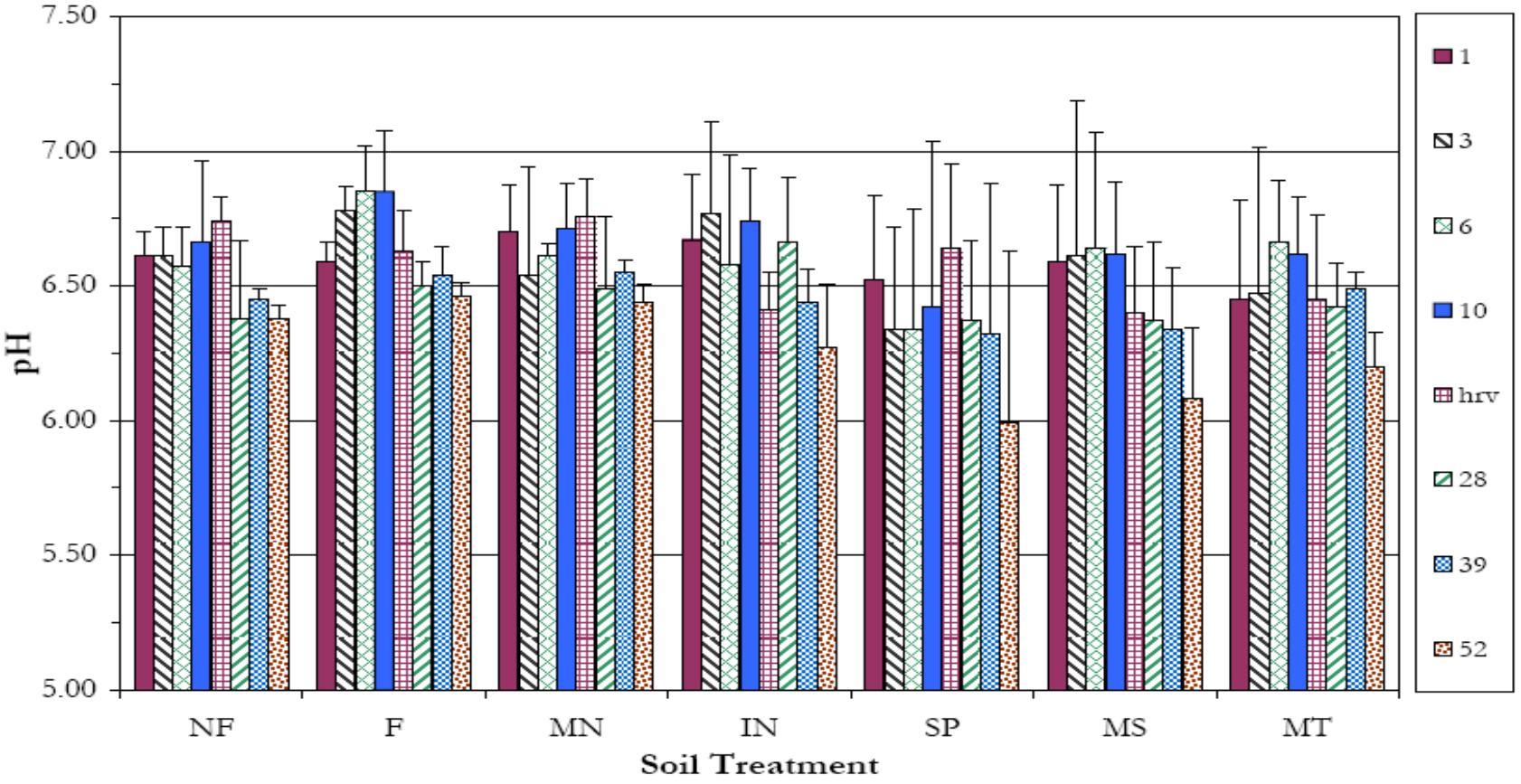


Figure 5.3-2: Soil pH measured over 12 month period following planting for each soil treatment: NF (unfertilized), F (fertilized), MN (manure), IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day). Time legend is in weeks; hrv=harvest. Error bars are 1 standard deviation

5.3.2 Soil Nutrients

5.3.2.1 Total Kjeldahl Nitrogen

Total Kjeldahl nitrogen (TKN) during the year is shown in Figure 5.3-3. The statistical analysis indicated that the effects treatment and time were significant (Pr_{MAX} = 0.015); row was not significant (Pr = 0.100). The interaction term, time*treatment, was statistically significant (Pr = 0.03), but time*row was not (Pr = 0.451). Additionally, TKN concentration at the 6 month sampling time was statistically lower than every other sampling time (Pr_{MAX} = 0.004). The qualification to the statistical analysis is that because of missing data, MN includes only 2 replicate plots instead of 3.

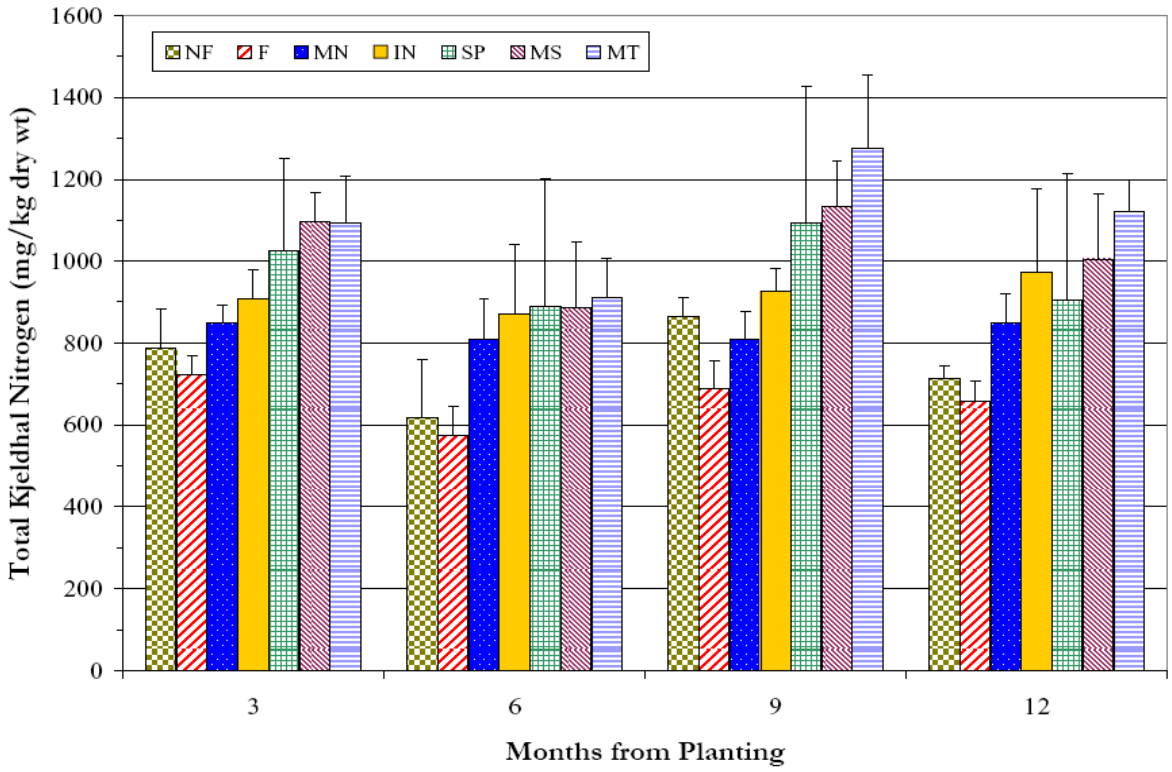


Figure 5.3-3: Total Kjeldahl nitrogen from harvest (3 months) until 12 months following planting for soil treatments NF (unfertilized, olive), F (fertilized, red), MN (manure, blue), IN (uncomposted, yellow), SP (19 day, green), MS (39 day, purple) and MT (103 day, aqua). Error bars are 1 standard deviation.

The trend for TKN at each sampling time generally corresponds to the OM loading rate of the organic amendments, which is shown in Table 4.2-5. The statistically significant ($\alpha = 0.05$) decrease in TKN at the six month sampling (December), which was one of the coldest months of the year (Appendix E), indicates that TKN levels were affected by microbial activity. Depressed microbial activity implies reduced microbial biomass (ie, organic nitrogen).

The positive controls consistently contained lower TKN levels than the negative controls. This may have been due to the higher microbial activity induced by the nitrogen fertilizer in the positive controls, resulting in mineralization of the soil organic matter (Broadbent, 1965), leading to lower TKN via nitrogenous gas losses and conversion of total ammoniacal nitrogen into nitrate.

5.3.2.2 Nitrate

Figure 5.3-4 shows the soil nitrate concentrations and Table 4.2-5 indicates the nitrate loading rates. The effects, treatment and row, were not statistically significant ($Pr_{MIN} = 0.057$); time was statistically significant ($Pr < 0.0001$). However, the interaction terms, time*treatment and time*row were not statistically significant ($Pr_{MIN} = 0.084$). The lack of treatment effects may have partly been due to the high sample variability, especially composts SP, MS and MT.

Soil nitrate levels are highly dependent upon environmental conditions since nitrate is very mobile. This was especially evident at the nine month (March) sampling time, when there was a reduction in nitrate levels for all treatments. This may have been caused by 1) higher microbial activity as the weather turned much warmer from the previous months, resulting in greater nitrogen assimilation than mineralization (see Chapter 6.3.2) and 2) higher precipitation (Appendix E) transporting nitrate deeper into the soil profile

5.3.2.3. Phosphorus

Soil concentrations of available phosphorus (Figure 5.3-5) from harvest (3 months) until 12 months after planting were measured values; initial concentrations were calculated based on phosphorus loading rates (Table 4.2-5). The statistical analysis pertains to the measured concentrations.

The effects of treatment ($Pr_{MAX} = 0.0001$) and row ($Pr = 0.050$) were statistically

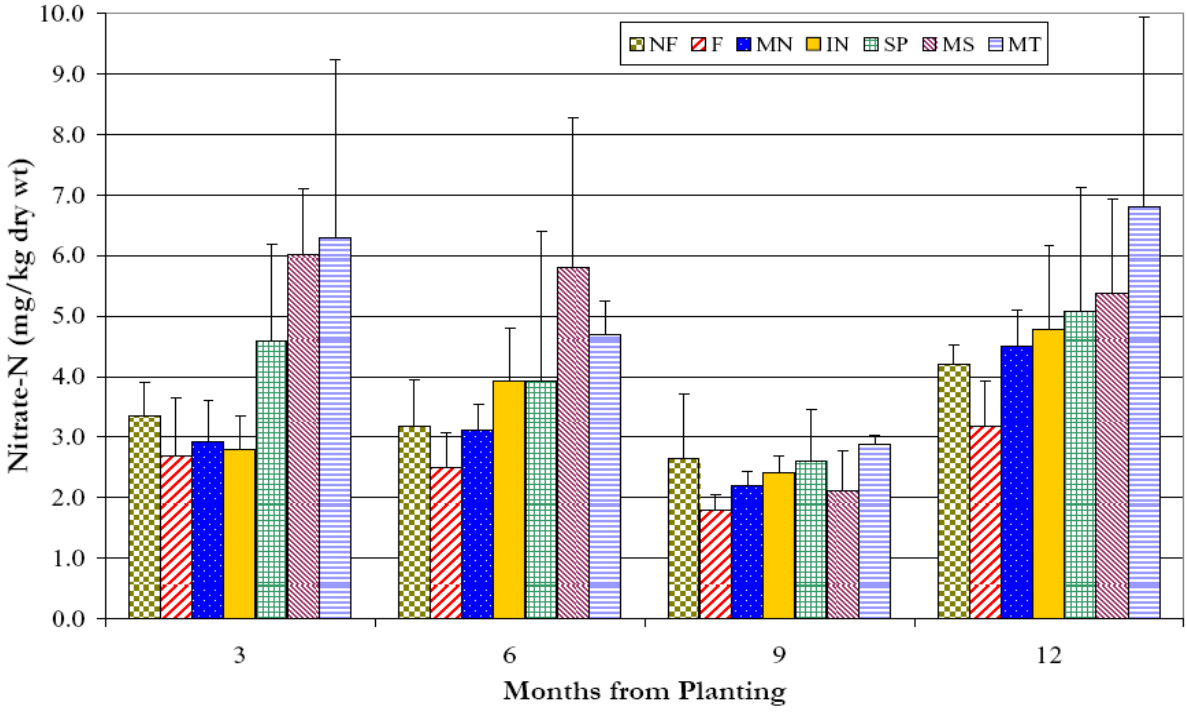
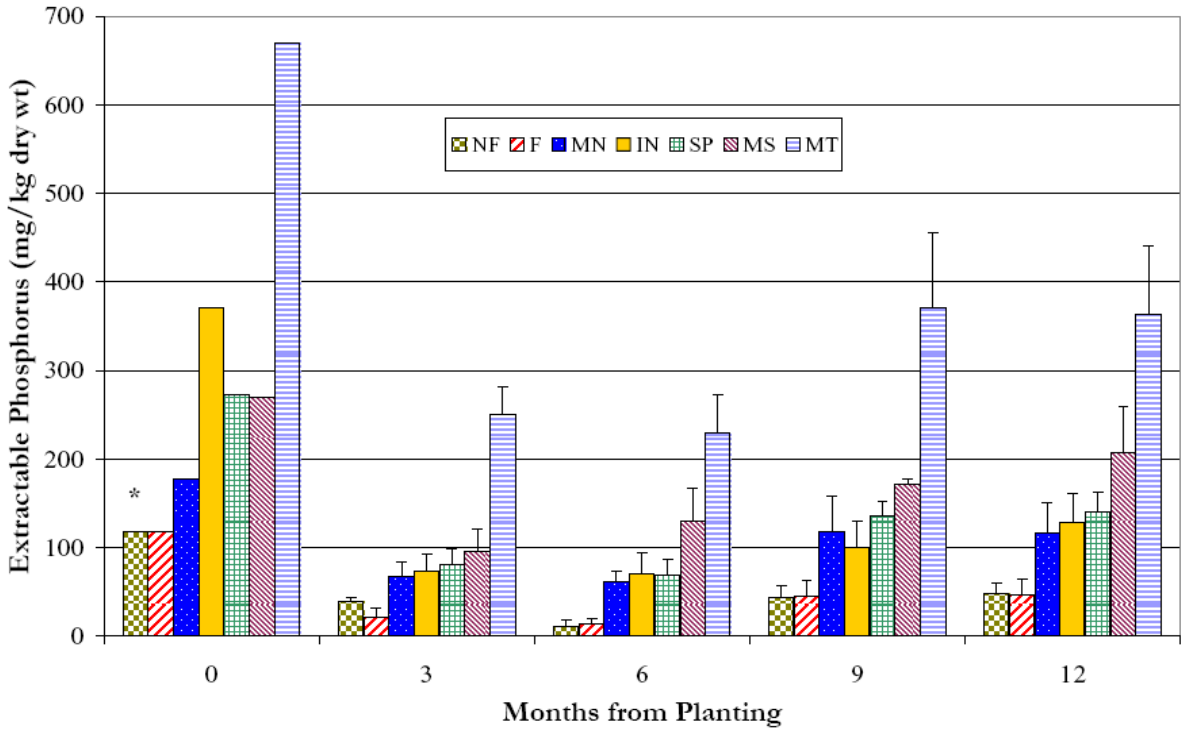


Figure 5.3-4: Nitrate concentrations from harvest (3 months) until 12 months following planting for soil treatments: NF (unfertilized, olive), F (fertilized, red), MN (manure, blue), IN (uncomposted, yellow), SP (19 day, green), MS (39 day, purple) and MT (103 day, aqua). Error bars are 1 standard deviation.

significant. Time was statistically significant (Pr_{MAX} = 0.0001). The interaction term, time*treatment was statistically significant (Pr = 0.011), but the interaction term, time*row was not (Pr = 0.313). Individual contrasts indicated that the 3 month time period was statistically equivalent to the 6 month period (Pr = 0.306), but statistically different from the 9 and 12 month periods (Pr_{MAX} = 0.0001). The 12 month sampling period was statistically different from the 3 and 6 month periods (Pr_{MAX} = 0.0001), but equivalent to the 9 month sampling period (Pr = 0.432).

The relative concentrations of phosphorus of the treated soils remained fairly consistent during the year (excluding initial levels). The concentrations in the non-fertilized and fertilized plots were similar, and lower than the organically amended soils, since no phosphate fertilizer



* Concentrations at sampling time 0 were calculated from amounts applied in amendments plus extant in soil (NCDA & CS soil test). BAE Environmental Analysis Laboratory determined available phosphorus in amendments using Mehlich 1 extractant; NCDA & CS determined soil phosphorus using Mehlich 3 extractant.

Figure 5.3-5: Mehlich 1 extractable phosphorus concentrations from harvest (3 months) until 12 months following planting for soil treatments: NF (unfertilized, olive), F (fertilized, red), MN (manure, blue), IN (uncomposted, yellow), SP (19 day, green), MS (39 day, purple) and MT (103 day, aqua). Error bars are 1 standard deviation.

was applied, only nitrogen and potassium. The trend for the organically amended soils appears to have been related to the phosphorus loading rates (Table 4.2-5) and the nature of the organic matter. The highest loading rate with MT corresponded to the greatest phosphorus concentration, although there was no clear relationship between the other amendment loading rates and soil phosphorus levels. Soil phosphorus levels have been reported to be related to the phosphorus loading rates (Adler et al., 2003; Schwartz et al.,2005). Alder et al. (2003) reported that weak acid (Mehlich 1) extractable soil phosphorus was not affected by compost age

(duration of composting) in compost amended soils, when the amendments were applied at equivalent phosphorus (Mehlich 1 extractable) loading rates. In the present study, the fact that MT with the highest phosphorus loading rate resulted in the greatest soil phosphorus levels, while MS with the lowest loading rate resulted in the second highest soil phosphorus concentrations indicates that amendment characteristics affected soil available phosphorus levels, contrary to Adler et al. (2003). As phosphorus has limited mobility in soil, this may indicate that MS contained more extractable (Mehlich 1) resistant forms of phosphorus (due to increased complexation) that were solubilized over time in the soil. MS was composted for a longer duration than SP or IN, which included an extended thermophilic phase. The longer composting time would have allowed more time for phosphorus complexation into less soluble forms; greater amounts of humified OM due to thermophilic decomposition (Chefetz et al., 1996; Hsu et al., 1999) may also have enhanced formation of less soluble organic complexes. While the decreases in available phosphorus during composting were attributed to both losses of labile phosphorus due to leachate losses and formation of less soluble complexes (Chapter 6.1.3.3), the higher levels of soil phosphorus in the MS and MT amended soils may indicate that the formation of less soluble complexes was the predominant mechanism.

The low loading rate of MN induced a similar phenomenon as MS. The soil phosphorus levels of the MN-amended soils were similar to the IN- and SP-amended soils, although the phosphorus loading rates for IN and SP were greater than MN. Therefore, the explanation for this phenomenon may be analogous to that for MS. Although MN was not composted, there were compositional differences between MN and the composts as MN did not contain bulking agent. Therefore, the manure may have contained higher amounts of less soluble phosphates that were not Mehlich 1 extractable, but solubilized over time in the soil.

5.3.2.4 Calcium and Magnesium

Figure 5.3-6 shows the calcium concentrations during the year. The effects, treatment and time were statistically significant (Pr_{MAX} = 0.0002); row was not statistically significant (Pr = 0.545). The interaction term, time*treatment was statistically significant (Pr = 0.002), but time*row was not (Pr = 0.148). Individual contrasts indicated that the 3 month sampling period was statistically different from all other time periods (Pr_{MAX} = 0.002); the 12 month sampling

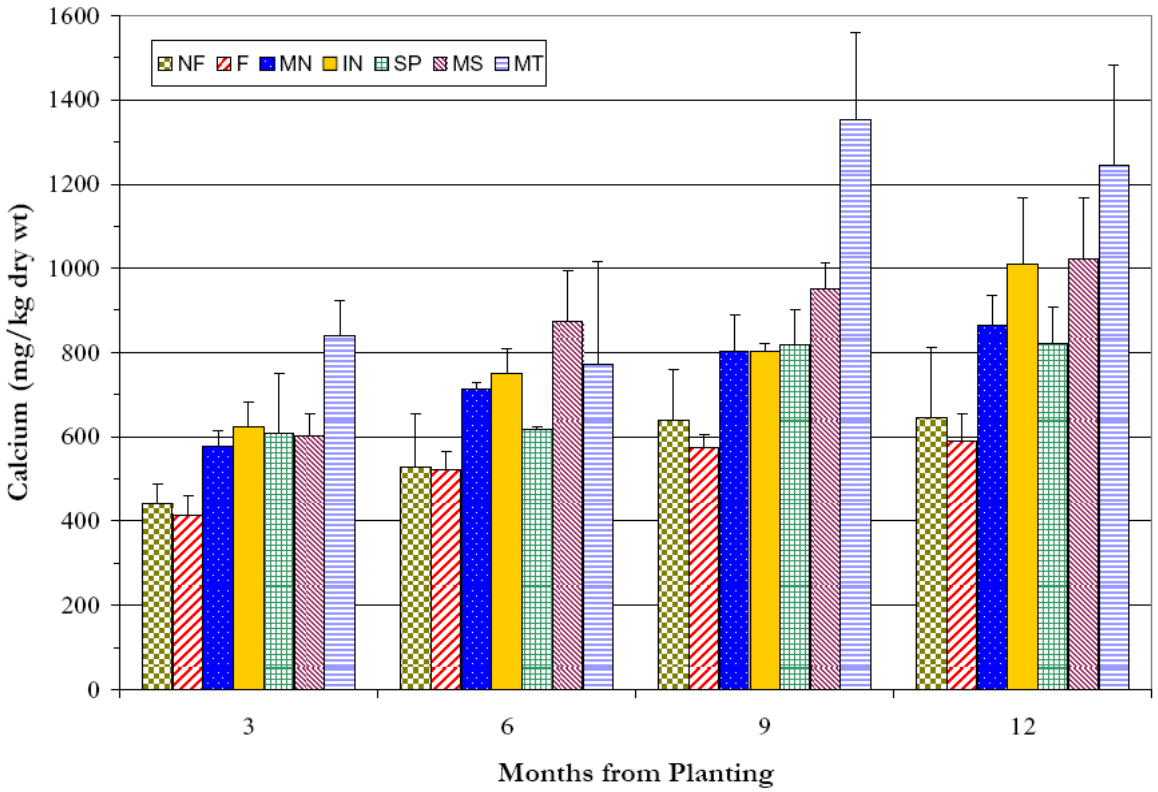


Figure 5.3-6: Calcium concentrations from harvest (3 months) until 12 months following planting for soil treatments**:** NF (unfertilized, olive), F (fertilized, red), MN (manure, blue), IN (uncomposted, yellow), SP (19 day, green), MS (39 day, purple) and MT (103 day, aqua). Error bars are 1 standard deviation.

period was different from the 3 and 6 month time periods (Pr_{MAX} = 0.002), but statistically equivalent to the 9 month period (Pr = 0.278).

Magnesium concentrations are shown in Figure 5.3-7. The main effects of treatment and row were not statistically significant (Pr_{MIN} = 0.146); time was statistically significant (Pr_{MAX} = 0.0001). The interaction term time*row statistically significant (Pr = 0.041), but time*treatment was not (Pr = 0.140). Individual contrasts indicated that the 3 month sampling period was statistically different from all other time periods (Pr_{MAX} = 0.006) and the 12 month sampling period was also different from all other time periods (Pr_{MAX} = 0.001).

There was a general trend of increasing calcium and magnesium concentrations as the year progressed (Figures 5.3-6 and 5.3-7, respectively). The similar trend of phosphorus levels

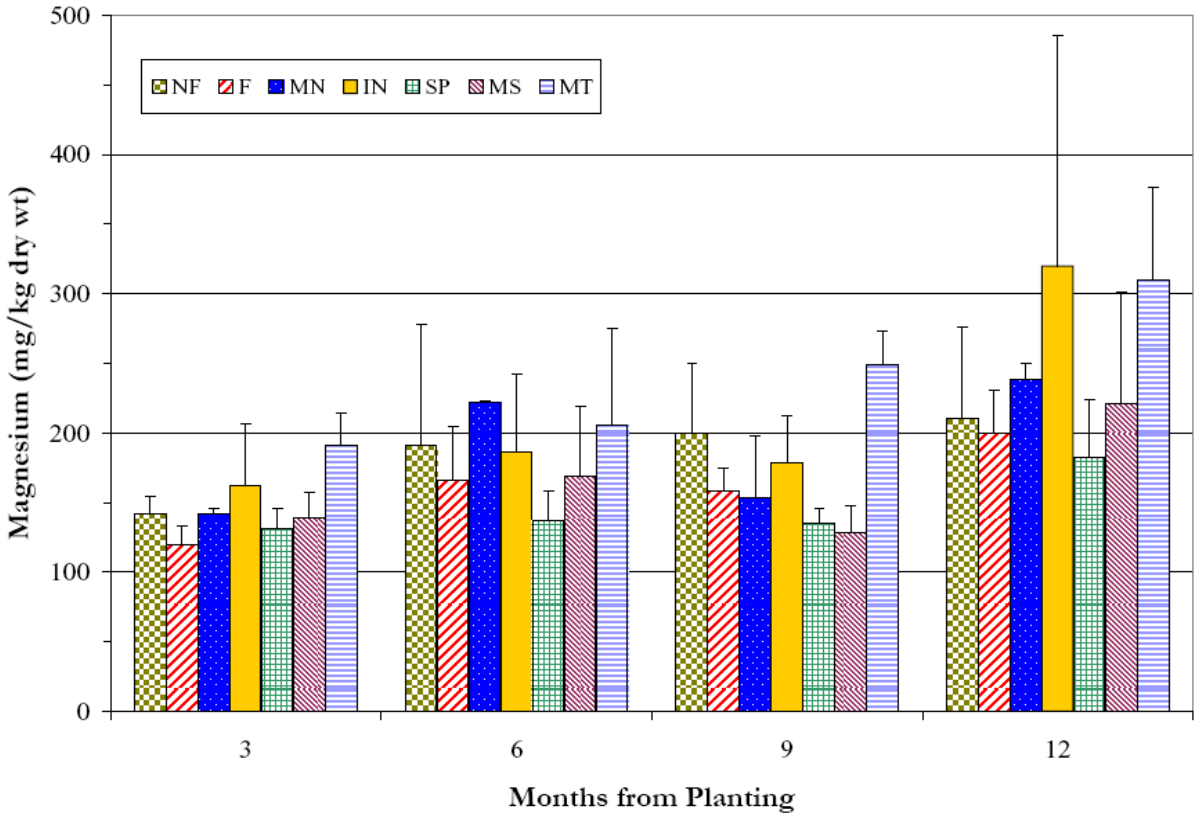


Figure 5.3-7: Magnesium concentrations from harvest (3 months) until 12 months following planting for soil treatments: NF (unfertilized, olive), F (fertilized, red), MN (manure, blue), IN (uncomposted, yellow), SP (19 day, green), MS (39 day, purple) and MT (103 day, aqua). Error bars are 1 standard deviation.

during the year (Figures 5.3-5) indicates that these increases may have been related to the general decrease in soil pH (Figure 5.3-2), increasing the solubilities of calcium and magnesium phosphates.

5.3.2.5 Copper and Zinc

Swine manure contains relatively high concentrations of copper and zinc because these metals are commonly added to swine feed due to their properties as antibiotics and growth promoters (Cromwell, 2001), as well as their ability to reduce obnoxious odors in wastes (Armstrong et al., 2000). Because of potential phytotoxicity to plants and the potential to be

detrimental to animals that forage on them, there is concern about long term land application of swine wastes. Based on the loading rates of the amendments (Table 4.2-5), increases in soil levels of copper and zinc were calculated (Table 5.3-1).

Table 5.3-1: Increases in soil concentrations of copper and zinc based on application of organic amendments compared to existing soil concentrations.

	MN	**IN**	**SP**	**MS**	**MT**	**Soil****
Copper*	2.5	3.3	3.3	3.7	10.2	6.0
Zinc*	12.6	11.3	16.3	20.2	51.5	6.6

* mg/kg soil dry wt.; based on a soil density of 1.28 g/cm^3 (Appendix B)
** based on values from soil test report (Appendix B) and Tucker et al. (1987)

In North Carolina, soil tests are mandated in fields receiving animal wastes (15A NCAC 02T.1300; NCDENR, 2003). Fields have to be removed from the application program if copper exceeds 60 mg/dm^3 (NCDA & CS soil test Cu Index = 3000) or zinc exceeds 120 mg/dm^3 (NCDA & CS soil test Zn Index = 3000) (NCDENR, 2003; NRCS 2004). Based on a soil density of 1.28 g/cm^3, these thresholds would be approximately 47 mg copper/kg soil and 94 mg zinc/kg soil. As copper and zinc are not readily mobile in soil (McBride, 1994), multiple applications, especially of the more composted feedstocks, could be problematic, based on the loading rates in the present study.

5.3.3 Physical Properties

5.3.3.1 Aggregate Stability

5.3.3.1.1 Data Analysis

Soil aggregate stability was calculated as the geometric mean diameter (GMD):

$$GMD = EXP\left\langle \sum \left[Wt \ \ logXt \ \right] / \sum Wt \right\rangle \qquad (5\text{-}3)$$

(legend follows on next page)

Wt = weight in each size class

Xt = mean diameter of corresponding intersize sieves

$\sum Wt$ = total sample weight

Four sieve sizes were used for determining aggregate stability: 1 mm, 500 μm, 250 μm and 105 μm. Aggregates less than the 105 μm size were not included in the calculations since it was not possible to determine the proportion of primary particles or organic matter.

In Figure 5.3-8, repeated measures of analysis indicated that the main effect, treatment, had no statistically significant effect (Pr = 0.384) on water aggregate stability, although row (Pr = 0.012) and time did (Pr_{MAX} = 0.0001). The interaction terms, time*treatment and time*row, were not statistically significant (Pr_{MIN} = 0.207). Individual contrasts indicated that the first sampling period (5 days following planting) was not necessarily statistically different (α = 0.05) from all other time periods, but the last sampling period (12 months) was different from every other time period.

5.3.3.1.2 Overview

Water (or wet) aggregate stability (WAS) is a measure of an aggregate's resistance to water induced stresses. As the value of WAS increases, the stability (or resistance) increases. The geometric mean diameter (GMD) and mean weight diameter (MWD) are the two numerical expressions recommended for expressing aggregate size distributions of soils (Kemper et al., 1986). Although MWD is common in the literature, Gardner (1956) indicated that in most soils the aggregate size distribution is log-normal rather than normal. Therefore, the wet aggregate stability data in the present study was presented as GMD.

Figure 5.3-8 shows that aggregate stability fluctuated during the one year of sampling (June '99 – June '00), following the same basic pattern irrespective of the soil treatment. Statistically, the effect of treatment was not significant (α = 0.05), nor did time have a significant effect on treatment, although the overall time effect was significant. Therefore, the temporal variations were not primarily due to the soil treatments, but climatic conditions. Consequently,

the seasonal fluctuations may have masked the variability due to the soil amendments. A similar conclusion was proffered by Alderfer (1950) concerning his investigation. In the present study, a similar fluctuation profile were observed in most of the treated soils: an increase at 2 months, followed by a decrease at harvest, then an increase in the warmer months of March (9 months) and June (12 months). However, these fluctuations may not have been statistically significant.

The stability of soil aggregates is a function of environmental conditions and soil biotic and abiotic factors. When amendments are added to the soil, characteristics of the amendments also have to be considered.

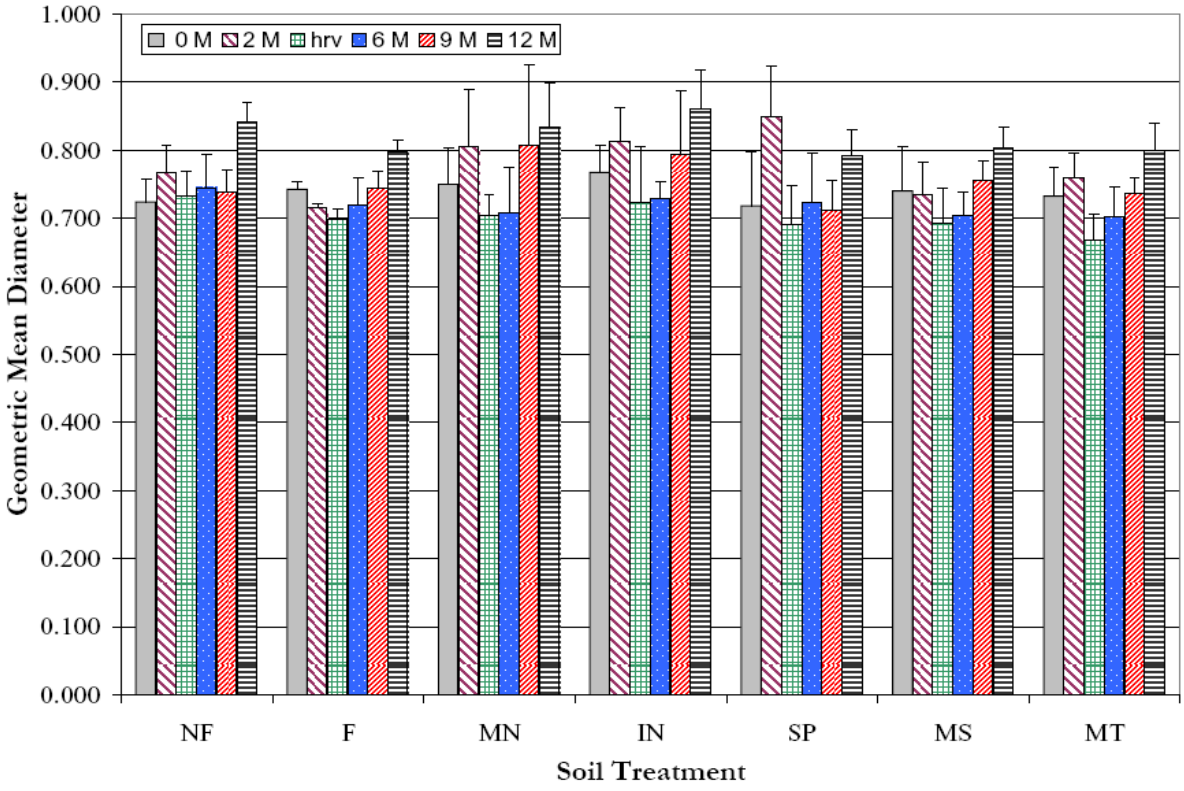


Figure 5.3-8: Soil aggregate stability, calculated as geometric mean diameter, for soil treatments: NF (unfertilized), F (fertilized), MN (manure), IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day). Colored columns indicate time from planting and are coded as follows: 0 months (grey), 2 months (purple), harvest (green), 6 months (blue), 9 months (red) and 12 months (black). Error bars are 1 standard deviation.

5.3.3.1.3 Climatic Factors

5.3.3.1.3.1 Temperature

Figure 5.3-8 shows indications of warm weather maxima, although the winter minima were not evident. Maxima were apparent at 2 months (August) and 12 months (June), although not for every treatment. Minima were observed at harvest, but not in the colder months. Temperature is a primary factor causing seasonal fluctuations since soil microbial activity increases as temperature increases (Avrahami et al, 2003; Bekku et al., 2003). Therefore, the sampling time in December (1999) would have been expected to reflect a nadir in aggregate stability since this month was one of the coldest of the year (Appendix E).

5.3.3.1.3.2 Precipitation

The sorghum was watered weekly if there was insufficient rainfall. Thus, for the growing season, water deficiency was not an issue. In September, the month of harvest, the precipitation accumulation was very high, over 16 inches (Appendices E & F); the 30-year average is about 5 inches (Appendix G). High water content of a soil may have resulted in hypoxic conditions that contributed to the lower aggregate stability (Avnimelech et al., 1988). For the remaining 9 months of soil sampling, except May 2000, the precipitation was within one standard deviation of the 30-year monthly average; the precipitation in May was about 1 ½ standard deviations lower than the 30 year average (Appendix G). Thus, no highly unusual precipitation accumulation occurred which would have contributed to atypical fluctuations in aggregate stability.

5.3.3.1.4 Soil Factors

5.3.3.1.4.1 Organic Amendments

In the present study, the results from the mineralization study indicated that the concentrations of inorganic nitrogen increased as the duration of composting increased (Figures 5.2-1 & 5.2-2), indicating that microbial activity was higher in the less composted feedstocks, supporting the hypothesis of Sela et al. (1998). Therefore, although the evolution of a slower release nutrient system during composting would indicate that the aggregating potential of the composts would have decreased in the order IN > SP > MS > MT, the very high microbial

activity (inferred by NNM) in the less composted feedstocks (IN and SP) appear to have reversed this trend. However, statistically, treatment effects were not significant ($\alpha = 0.05$).

In the present study, composts MS and MT were composted for the longest time and both progressed through a complete thermophilic phase and therefore would have tended to have had less available nutrients than MN, IN, and SP, which were not composted (MN, IN) or allowed to proceed through a full thermophilic phase (SP) (Bernal et al., 1998c; Benito et al., 2005). Therefore, the WAS induced by MS and MT would have been lower (and more similar), than the WAS induced by the composts that did not undergo (full) thermophilic decomposition. The lower nutrient levels plus a slower nutrient release with increased composting time would have hindered MS and MT from catalyzing low oxygen conditions (Figure 5.2-2). Additionally, if the less composted amendments (MN, IN, SP) had induced lower redox conditions (Miller et al., 1985; Katayama et al., 1987), instead of enhancing WAS, the reverse might have occurred to varying degrees. The latter phenomenon may have contributed to the lack of treatment effects.

Compared to the manure (MN), IN should have had a lower aggregating potential due to the relatively large amount of recalcitrant OM (lignin-like fraction) comprising the wood chips/leaves bulking agent, which reduces decomposition (Chapter 5.2.3.4). Additionally, the particle sizes of the wood chips and leaves were much larger than that of the manure. The much larger volume:surface area ratio of the bulking agent, especially the wood chips, would have tended to decrease decomposition rates. However, WAS for the MN and IN amended soils were similar.

To maximize aggregate stability, Avnimelech et al. (1989) recommended a C:N range of 20-40 for organic soil amendments, accounting for responses from different organic amendments, including changes in bulk density, modulus of rupture, and concentrations of polysaccharides and polyuronides. This C:N range resulted in high aggregate stability that corresponded to high levels of microbial carbohydrates (considered to be a measure of particle binding potential). The recommended C:N range corresponds to the nutrient ratio that optimizes microbial efficiency (see Chapter 2.2.4.1). Based on microbial activity, Dąbek-Szreniawska et al. (2004) hypothesized that stabilization of water-stable aggregates is enhanced by microbial metabolic efficiency. In the present study, the C:N ratios of MN and IN treatments, when field applied were within the C:N range of 20-40, while the SP, MS and MT treatments

were below the range (Figure 5.1-6). From the inference of Avnimelech et al. (1989), amendments MN and IN should have contributed more to the enhancement of soil aggregation than composts SP, MS and MT. However, this was not the case.

The previous discussion concerned biotic amendment characteristics that could have promoted WAS in the present study. However, as soil treatment was not a statistically significant ($\alpha = 0.05$) variable, abiotic factors may have been pivotal in controlling WAS.

The presence of multivalent cations, such as copper and zinc in swine manure may have been factors regulating aggregate formation. The charge on OM is pH dependent, and at normal soil pH (~3-8), OM contains primarily negatively charged functional groups of which carboxyl and phenolic/alcoholic ligands are the most important (Senesi et al., 1999; Tan, 2003) and these ligands appear to be the primary moieties involved in aggregation (Harris et al., 1973). Due to the negative charges, multivalent cations readily complex with OM. Copper forms the most stable complexes with OM of any divalent cation, and can form chelates (McBride, 1994; Stevenson, 1994). Chelation is an especially stable form of bonding, where multiple bonds form between polyvalent cations and associated functional groups of a substrate, reducing binding sites on the OM. In the case of non-rigid OM, the crosslinking of functional groups may result in more condensed, rigid molecules, which tend to be more resistant to chemical and biological attack, especially when bonded to clays, and therefore may provide slower degradation and thus, release of nutrients (Muneer et al., 1989a & c). The reduced number of potential binding sites on the OM and availability of nutrients, which would reduce microbial activity and consequently the production of extracellular (binding) carbohydrates, would tend to diminish the potential for enhancing aggregation. However, Robert et al. (1992) suggested that calcium cations may crosslink anionic polysaccharides, creating gels that would enhance aggregation. If this were so, then copper would be an even stronger promoter of aggregation.

It appears that two opposing transformations occurred, involving cation-OM interactions. In the present study, given the lack of treatment effects on aggregate stability, the presence of calcium, zinc and especially copper, in the organic soil amendments, may have contributed more to the inhibition of WAS than enhancement.

5.3.3.1.4.2 Application Rates

The fact that overall the effect of treatment on WAS was not statistically significant ($\alpha = 0.05$) may have been partially due to low application rates of the soil amendments. OM is a source of nutrients for microbes and soil microbial activity is positively correlated with organic amendment loading rate (Tester et al., 1977; Nishio et al., 1980; Saviozzi et al., 1993; Pascual et al., 1998). However, increased application rates do not necessarily translate into enhanced WAS. In short term studies (up to about 1 1/2 years), Guidi et al. (1989) from the results of a non-calcitic soil, and Pagliai et al. (1981) reported positive correlations between organic amendment application rates and WAS, although the differences in WAS were not necessarily statistically significant. In long term experiments (10 years or more), Ibrahim et al. (1999) reported increases in WAS with increasing compost application rates, although no statistical analysis was performed due to the lack of replication; Albiach et al. (2001b) reported no statistically significant changes with increasing loading rates of wastewater sludges. However, although the latter authors provided limited soil characteristics, the high soil pH of 8.2 (Albiach et al., 2000) indicated a calcareous soil, which may have depressed OM stimuli (see below, Chapter 5.3.3.1.8).

The results of Guidi et al. (1988) also provide evidence of a minimum response threshold. The authors amended a calcitic soil with three rates of compost (C1, C2, and C3), manure and inorganic fertilizer. The fertilizer, manure and C2 were applied at 200 kg/ha, C3 at 300 kg/ha and C1 at 100 kg/ha, based on available nitrogen. Corn was planted in May and harvested in November. For all treatments, wet aggregate stability peaked in September and was lowest in November. Except for the compost at the lowest loading rate, the maxima were statistically significantly different from the other 2 sampling periods (June and November). For the lowest compost loading rate, there were no statistically significant differences between sampling periods (May, June and November). In addition, the maximum for C1 was much lower than for all other treatment maxima, which were similar, although any differences were not statistically significant. The lower C1 maximum may indicate that a minimum OM loading rate exists to overcome abiotic factors, in this case calcareous soil.

In the present study, the organic amendments were added based on the nitrogen requirements of the plants and the projected nitrogen mineralization rates of the amendments. Since the mineralization rates may have been overestimated, based on the results of the nitrogen

mineralization study, lower amounts of compost may have been applied, with the exception of MT. Consequently, the effectiveness of the amendments may have been reduced.

5.3.3.1.4.3 Abiotic Soil factors

Texture and mineralogy are important factors in how a soil responds to organic amendments. In the present study, the soil was a fine, sandy loam and therefore, the clay content was less than 35%, as the soil was not classified as clayey. Being classified as kaolinitic, indicates that at least 50% of the clay was comprised of kaolin. Kaolin has a 1:1 layered structure, and thus a low cation exchange capacity (CEC). The statistical analysis, indicating that the effect of soil treatment on WAS was not statistically significant, appears to confirm that low reactivity soils have a reduced response to OM (Oades et al., 1991; Six et al., 2000a & b).

Calcium may mute the aggregating potential of OM (Stefanson, 1968 & 1971; Guidi et al., 1988) and primarily stabilizes micro-aggregates ($\leq$ 250 μm) (Dąbek-Szreniawska et al, 1977; Muneer et al., 1989a, b & c). Stefanson (1968 & 1971) attributed the lack of seasonal variation in aggregate stability to the high calcium carbonate content of the soil, based on measurements of micro-aggregation Similarly, although not using the conventional demarcation of 250 μm to denote micro-aggregates, Blackman (1992) reported little seasonal variation in aggregates less than 500 μm, but greater variation in aggregates greater than 500 μm in a calcareous soil. Guidi et al. (1988) reported a lack of statistical differences between WAS from a calcareous soil amended with mineral fertilizer, manure or compost, based on micro-aggregation. The results of Rennie et al. (1954) indicated that lime increased WAS, although the measurements were not based on micro-aggregates per se. They were based on a composite index of aggregates 0.10 – 6.3 mm.

In the present study, calcium constituted 61% of the soil CEC (Appendix B), although this number was conservative since the soil sampling occurred prior to lime addition. Calcitic lime was applied at a rate of 2 t/ac, about 3 months prior to planting, and this loading would have added approximately 10% to the total calcium concentration. For determining WAS in the present study, a composite index was calculated using aggregates 2 mm (initial maximum size) to 105 μm (minimum sieve size). Consequently, the impact of calcium would have been included in WAS measurements. Therefore, it appears that the low CEC of the kaolinitic soil plus the

presence of calcium cations, were factors in suppressing the effects of OM on aggregate stability in the present study.

5.3.3.1.4.4 Tillage

Initial speculation was that tillage might have diminished aggregate stability, contributing to the differences between the initial and subsequent sampling time (2 months). The final disking of the field occurred on the day of sowing, 5 days prior to the first sampling. The effects of tillage are multifold. Tillage disrupts soil, physically breaking up weakly held aggregates, and exposing more of the soil to erosion potential (Brady, 1990). More of the soil is also exposed to oxygen and precipitation, both of which stimulate microbial activity (Balesdent et al., 2000) and consequently aggregating potential. Thus, there are opposing aggregating mechanisms. While long term cultivation ($\geq$ 2 years) indicates that tillage reduces OM, microbial activity and water aggregate stability, compared to no-till soil management (Linn et al., 1984a & b; Bruce et al., 1990; Whalen et al., 2003), short term effects do not necessarily mimic long term effects.

Measurements taken two days after rototilling, on soils that had been air dried prior to wet sieving, showed decreased WAS, although by 7 days following tillage the WAS had returned to the approximate pre-tillage levels (Bullock et al., 1988). Similarly, Plante et al. (2002) observed that approximately nine days following tillage, the WAS was at pre-tillage levels. Bullock et al. (1988) also indicated that rototilling was a more severe disruption of soil than either plowing or disking; disking was the cultivation method used in the present study. Therefore, in the present study, although the WAS at the initial sampling time was statistically less ($\alpha = 0.05$) than at the following sampling (2 months) (Appendix H), the disruptive action of tillage was probably not a factor affecting wet aggregate stability.

5.3.3.1.4.5 Plants

In the present study, aggregate stability trends appear to have been dependent upon the cycles of plant growth in the field. Heatherly (1975) reported that root density of grain sorghum increased until 5 weeks after planting, remained at the maximum until 2 weeks prior to maturity, then declined rapidly afterwards. This root density temporal profile is similar to the aggregate stability trend during the growing season in the present study. Figure 5.3-8 shows that 2 months

following planting, there was a general increase in WAS, followed by a decrease in all treatment plots at harvest. Therefore, a decrease in the third month (harvest) would not be unexpected, and is similar to the results of Guidi et al. (1988), who reported the lowest WAS of the growing season at harvest. In the present study, the subsequent increases in March and June, the nine month and 12 month sampling, respectively, indicate warmer months, and thus increased microbial activity, which would have been sustained by nutrients, in part, by slowly decomposing sorghum roots following harvest (Lynch et al., l980), and weed growth. The field was only weeded intermittently after harvest and thus the warmer months would have seen weed growth, and possibly an enhancement of WAS due to root growth.

5.3.3.1.4.6 Size Matters

Stable aggregates are necessary to maintain pore structure, and pore sizes are important factors in water availability, water and air transmission characteristics and root growth. Certain soil particle sizes are associated with forming particular size pores (Oades, 1984; Boix-Fayos et al., 2001). Since a distribution of pore sizes is necessary to accommodate soil characteristics that enhance plant growth, a distribution of particle sizes is necessary to optimize the pore size distribution.

Several investigators have attempted to quantify optimum soil aggregation. Oades (1984) suggested that in arable soils, most of the clay fraction should be flocculated into micro-aggregates (< 250 μm) and the majority of the macro-aggregates (> 250 μm) should be 1 to 10 mm in diameter. Vepraskas et al. (1990) concluded that corn root growth was greatly restricted when the MWD of the soil was less than/equal to 1.2 mm, but much less restricted with a MWD of greater than 2. Schneider et al. (1985) reported that corn emergence was depressed in soils with GMD of 11.1 and 0.5, but adequate from 1.0 to 6.8, the range between the extremes. In the present study, the range of aggregate stability was approximately 0.65 to 0.85 (GMD), which was determined by a wet sieving method. In the studies of Vepraskas et al. (1990) and Schneider et al. (1985), the aggregate size distributions were determined by dry sieving. However, aggregate size per se, does not necessarily confer aggregate stability (Dormaar, 1983; Angers et al., 1988; Sort et al., 1999; León-González et al., 2000).

Comparing the results of different investigations is often difficult because of the variety

of methods used, and the size of aggregates analyzed. The latter is especially pertinent since macro-aggregate (> 250 μm) and micro-aggregate (< 250 μm) stability is inversely related (Muneer et al., 1989a; Perfect et al., 1990b). Determining aggregate stability, using different procedures, has often resulted in disparate rankings for the same soils (Williams et al., 1966; Pojasok et al., 1990; Beare et al., 1993; Amézketa et al., 1996).

5.3.3.1.5 Sample Variability

Part of the reason for "unobservable" soil response in the present study, may have been due to the high sample variability that is evident in Figure 5.3-8. A similar conclusion was offered by Plante et al. (2002) in their investigation. The authors amended 2 different soils with inorganic nitrogen either with or without straw, for 2 successive years. Over the duration of the experiment, straw additions significantly increased WAS in the Malmo (smectitic) soil, but not in the Breton (mica/amorphous minerals, Howitt et al., 1985) soil. The authors pointed out that in both soils, the increase was similar, but that in the Breton soil, the increase was actually proportionally greater. They concluded that the higher variability of the Breton samples resulted in lack of statistical significance.

In the present study, the harvest, and thus soil sampling, occurred over about 3 weeks, although the majority of plots were harvested within a 6 day period. The standard deviations of each treatment during the harvest were similar to the other sampling times, when the samples were collected on one day. This indicates that the wet aggregate stability was constant over many weeks, at least during the harvest period.

5.3.3.1.6 Short Term Investigations

Although there was a lack of treatment effects in the present study, OM additions to soil have produced increases in WAS in short term studies (< approximately 1 year). Statistically significant increases in WAS have been reported three months following application of composted municipal wastewater residuals (Sort et al., 1999) and twelve months following application of composted and uncomposted municipal refuse (Caravaca et al., 2001). Pagliai et al. (1981) amended soil with different uncomposted or composted feedstocks. One month following application, increases in WAS were measured in all amended soils, although the

increases were not necessarily statistically significant compared to the unamended control. Subsequent measurements at 3 and 12 months following amendment application produced similar results with more of the amendments producing statistically significant increases in WAS as time increased.

5.3.3.1.7 Summary of Conclusions

Soil amendments induced few changes in WAS, although these results were not necessarily inconsistent with other short term investigations. Abiotic factors appear to have been the controlling mechanisms of the diminished soil response to the organic amendments. The high kaolin content of the soil, which resulted in a relatively low CEC, seems to have been the primary determinant. Calcium (present in the soil and added with liming) and zinc and copper (from the organic amendments), may have reduced the effectiveness of the OM in increasing WAS by competing with organic moieties for binding sites on clay particles, and by chelating with the OM, reducing the number of potential binding sites on the OM. Other contributing factors may have been suboptimum application rates of the organic soil amendments as well as the high variability of the measurements, which may have masked differences in soil responses.

5.3.3.2 Bulk Density

Figure 5.3-9 is the soil bulk density measured four times during the year. Time and treatment were statistically significant ($Pr_{MAX} = 0.003$); row was not statistically significant ($Pr = 0.725$). The interaction terms, time*treatment and time*row, were not statistically significant ($Pr_{MIN} = 0.279$). One degree of freedom contrasts indicated that the initial time was statistically significantly lower than the other sampling times ($Pr_{MAX} = 0.0001$). However, excluding the initial sampling time produced similar statistical results.

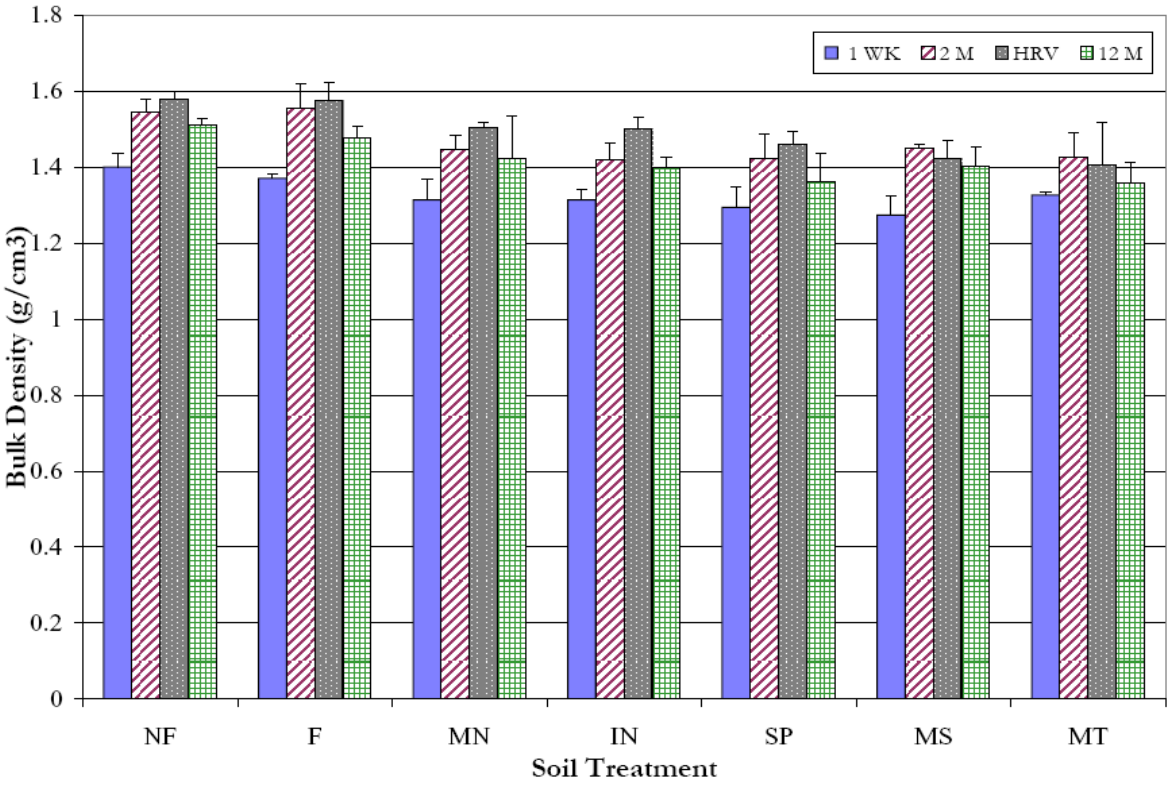


Figure 5.3-9: Soil bulk density for soil treatments**:** NF (unfertilized), F (fertilized), MN (manure), IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day). Colored columns indicate time from planting and are coded as follows:1 week (blue), 2 months (purple), harvest (grey) and 12 months (green). Error bars are 1 standard deviation.

Overall, the organically amended soils lowered the soil bulk density, as the effect of treatment was statistically significant ($\alpha = 0.05$). Soil bulk density is reduced by mixing a lower density fraction (OM) with a higher density fraction (soil) and by the changes in soil structure that are induced by enhanced microbial activity due to the OM (Nishio et al., 1983). Soil structure often improves by increasing particle aggregation, which can alter particle packing and affect total porosity (Pagliai et al., 1981; Monreal et al., 1997). Wet aggregate stability and soil bulk density are inversely correlated (Coote et al., 1988; García-Orenes et al., 2005; Ray et al., 2006), although Schneider et al. (1985) reported a parabola-like relationship (concave up) between aggregate size distribution and bulk density, as the aggregate size distribution increased.

The present author analyzed the data of Pagliai et al. (1981) and found a low, positive correlation (Pearson correlation coefficient = 0.33) between their water (aggregate) stability index and total porosity, over all soil treatments (manure, municipal sludge, composted sludge and negative control). In the present study, there was a low, negative correlation (Pearson correlation coefficient = ⁻ 0.42) between WAS and bulk density. The correlation included all of the soil treatments for the common sampling times of two and twelve months. (At 12 months, samples for both characterizations were collected on the same day; for the 2 month time period, there was a 10 day differential between the samples collected for bulk density and WAS.) The common initial sampling time was excluded due to the possible confounding effects of soil tillage. The low correlation was not unexpected, since soil aggregation was minimally affected by the soil amendments.

In the present study, the increase in bulk density following the initial sampling event may have been due to soil tillage at the beginning of the field trial and the presence of stones in the soil. The first sampling occurred one week following application of the soil amendments, which were disked into the soil. The tillage would have disrupted the soil and temporarily decreased the bulk density. Over time, the soil would have settled, increasing the density (Osunbitan et al., 2005). The rockiness of the soil may also have contributed to the higher bulk densities. Two and often three attempts were necessary to obtain each acceptable soil sample, because the stones hindered the soil corer from being driven into the soil. Even the acceptable samples did not necessarily preclude the use hard pounding. Consequently, the density of soil may have been artificially increased.

In short term experiments (less than ~ 1 year) reductions in soil bulk density following application of uncomposted or composted OM often occur (Pagliai et al., 1981; Mbagwu et al., 1989; Aggelides et al., 2000; Yuksel et al., 2004), although invariances have also been reported (King et al., 1969; Gonzalez et al., 2002). However, Guerrero et al. (2000) reported municipal solid waste compost increased soil bulk density, although the increases were not necessarily statistically significant. The effects on bulk density are dependent upon organic matter and soil characteristics, as well as plant species (Khaleel et al., 1981; Gonzalez et al., 2002; Chapter 5.3.3.1).

5.3.3.3 Penetrometer Resistance

Figure 5.3-10 shows the penetrometer resistance of the soil from amendment application until several weeks following the last harvested plot. Statistical analysis indicated that the variable time was statistically significant (Pr_{MAX} = 0.0001), whereas effects of treatment and row were not significant (Pr_{MIN} = 0.504). The interaction terms, time*treatment and time*row were not statistically significant (Pr_{MIN} = 0.6222). Using one degree of freedom contrasts, analysis indicated the initial time (5 days) was statistically greater than every other time period (Pr_{MAX} = 0.0001). Excluding the initial sampling time from the analysis did not change the results.

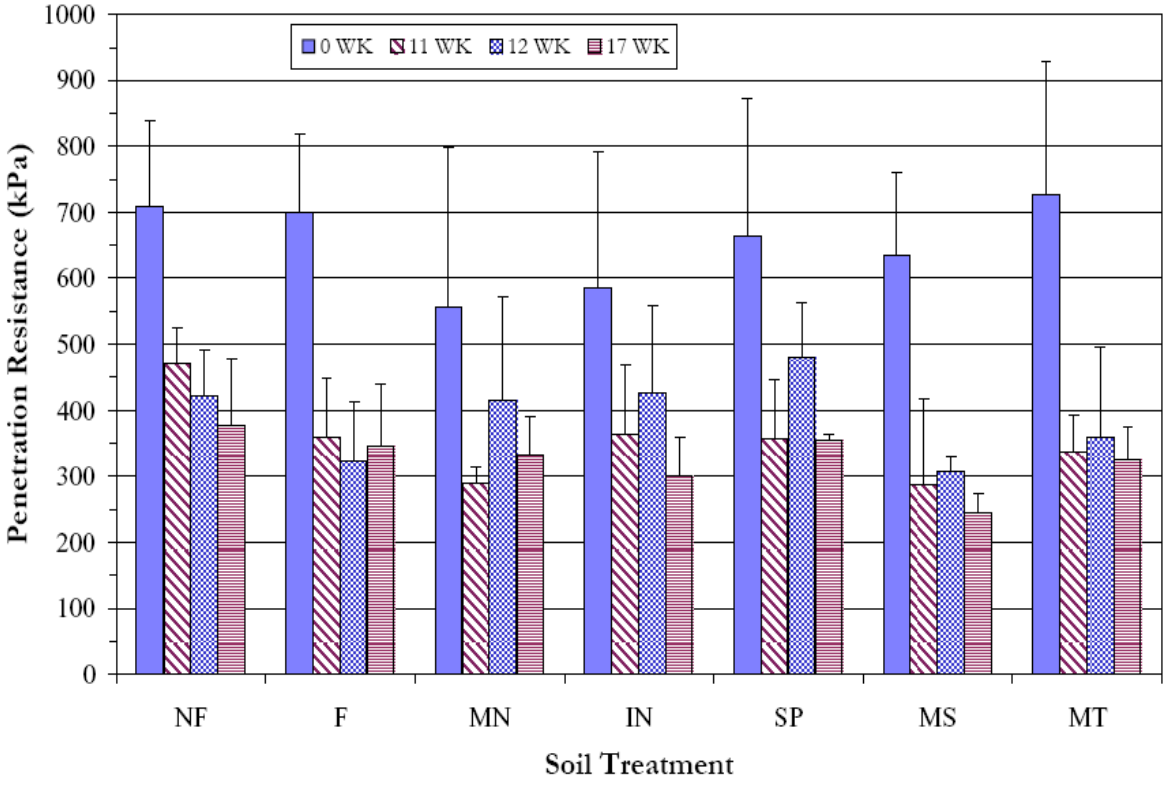


Figure 5.3-10: Soil penetration resistance for soil treatments: NF (unfertilized), F (fertilized), MN (manure), IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day). Colored columns indicate time from planting and are coded as follows: 0 weeks (solid blue), 11 weeks (red angled stripes), 12 weeks (checkered blue) and 17 weeks (red horizontal stripes). Error bars are 1 standard deviation.

The initially high resistance decreased significantly about 10 weeks later. Resistance measurements were taken in the vehicle traffic rows between sorghum rows. Following amendment application, a planter vehicle made one pass for seeding. The first measurements were taken 5 days following planting, and the higher standard deviations indicate the greater nonuniformity of the soil compared to later measurements. Compared to subsequent measurements, soil compaction from the planter tires may have resulted in higher penetration resistance (Motavalli et al., 2003), as the measurements were taken so soon after seeding.

Additionally, there is an inverse relationship between soil moisture and penetration resistance (Aggelides et al., 2000). The soil was drier during the first measurement period than the subsequent ones at 11, 12 and 17 weeks (Figure 5.3-11), which may have contributed to the

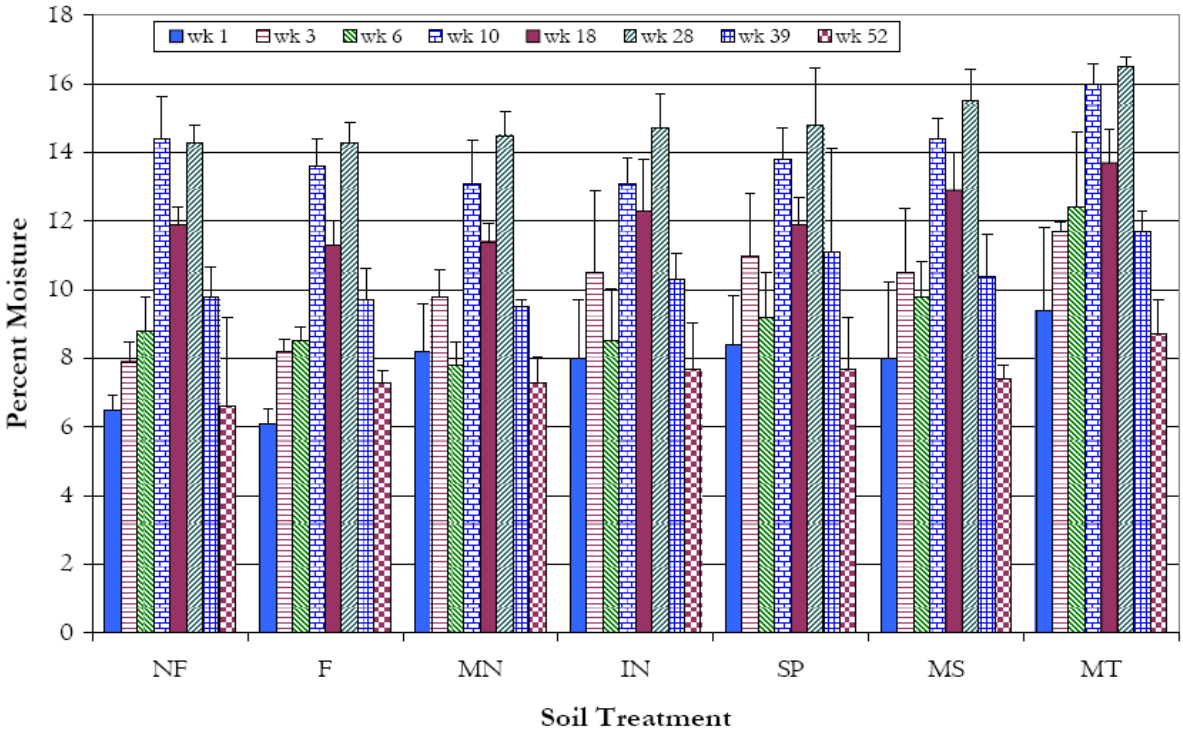


Figure 5.3-11: Soil moisture during a 12 month period. The time legend indicates weeks following planting. Error bars are one standard deviation.

high, initial impedance values. Schneider et al. (1985) reported a complex relationship between penetration resistance and aggregate size distribution, as soil moisture content was a confounding factor. However, aggregate size distribution does not necessarily equate with WAS (Dormaar, 1983; Angers et al., 1988; Sort et al., 1999; León-González et al., 2000).

Similar to the lack of treatment effect in the present study, Bazzoffi et al. (2000) reported no statistically significant difference ($\alpha = 0.05$) in penetrometer resistance between MSW compost amended and unamended soils in the first year of their study. Motavalli et al. (2003) also reported no statistically significant differences ($\alpha = 0.10$) between turkey litter amended and unamended soil from 1-3 years after application in uncompacted soil. Conversely, MSW compost reduced soil impedance compared to unamended soils 2 months following amendment application, although no statistical comparisons were made (Avnimelech et al., 1990).

5.3.3.4 Water Holding Capacity

Soil water holding capacity (WHC) at field capacity is defined as the amount of water retained by soil under the influence of only gravity and is often measured at (-0.01 MPa). When plants become permanently wilted due to insufficient soil water, "wilting point" has been attained. WHC at wilting point is measured at -1.5 MPa. Plant available water (PAW) is the calculated difference between field capacity and wilting point, and indicates the amount of soil water that is readily available to plants. Maximizing PAW is desirable.

In the present study, at both field capacity (Figure 5.3-12) and wilting point (Figure 5.3-13), the effects of treatment were not significant ($Pr_{MIN} = 0.093$), nor were row ($Pr_{MIN} = 0.052$), although the interaction term, treatment*row was significant ($Pr_{MAX} = 0.0001$). The overall effect of time was statistically significant ($Pr_{MAX} = 0.0001$). At field capacity, the interaction term, time*treatment, was not statistically significant ($Pr = 0.167$), while at wilting point it was ($Pr = 0.0002$); at wilting point at every time period, treatment was statistically significant ($Pr_{MAX} = 0.0001$).

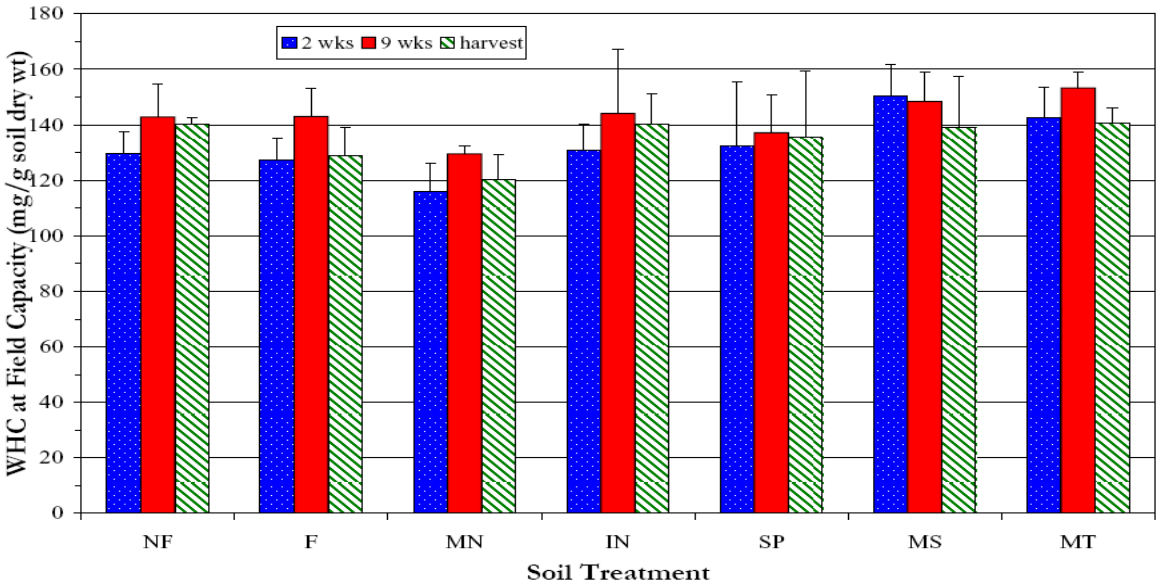


Figure 5.3-12: Water holding capacity at field capacity (–0.01 MPa) for soil treatments: NF (unfertilized), F (fertilized), MN (manure), IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day). Colored columns indicate time from planting and are coded as follows: 2 weeks (blue textured), 9 weeks (red) and harvest (green striped). Error bars are 1 standard deviation.

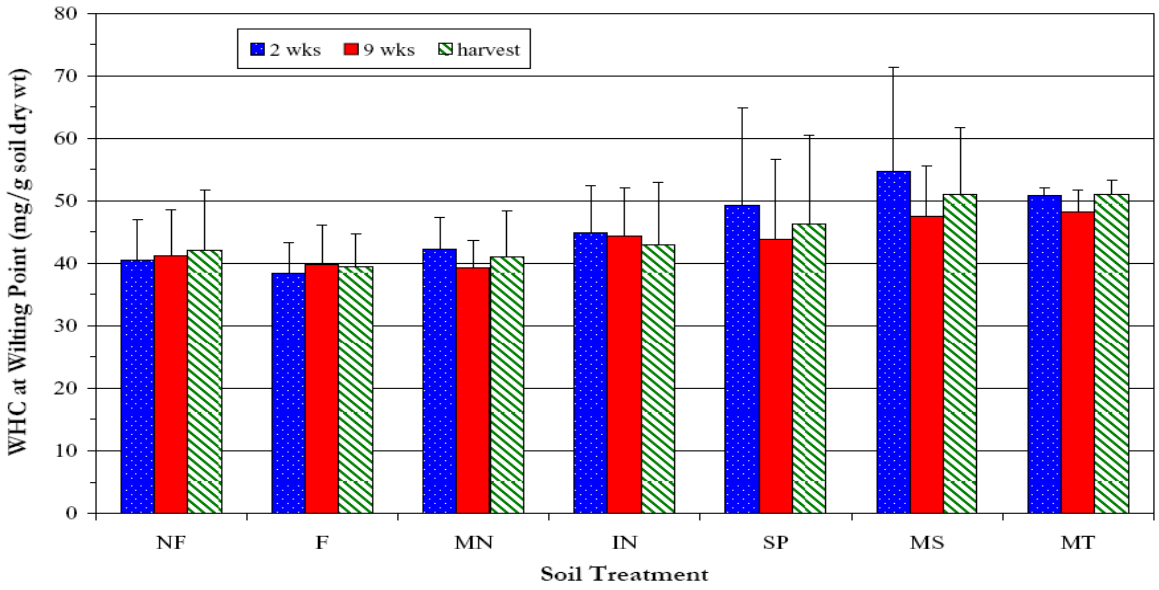


Figure 5.3-13: Water holding capacity at wilting point (–1.5 MPa) for soil treatments: NF (unfertilized), F (fertilized), MN (manure), IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day). Colored columns indicate time from planting and are coded as follows: 2 weeks (blue textured), 9 weeks (red) and harvest (green striped). Error bars are 1 standard deviation.

For plant available water (Figure 5.3-14), the effect of treatment, was statistically significant (Pr = 0.0004), although the effect of row was not (Pr = 0.271); the interaction term, row*treatment, was not statistically significant (Pr = 0.970). The variable, time, was statistically significant (Pr = 0.0002), but the interaction term, time*treatment, was not (Pr = 0.802). MN was statistically less than all other treatments, except SP (Pr =0.127). All other treatments, except MN, were statistically equivalent (Pr_{MIN} = 0.063).

The water holding capacity (WHC) at field capacity and wilting points were not statistically affected by soil treatment (α = 0.05). Plant available water (PAW), however, was affected by treatment, with MN being statistically lower than every other treatment, except SP; all other treatments were statistically equivalent. The almost total lack of treatment effects is

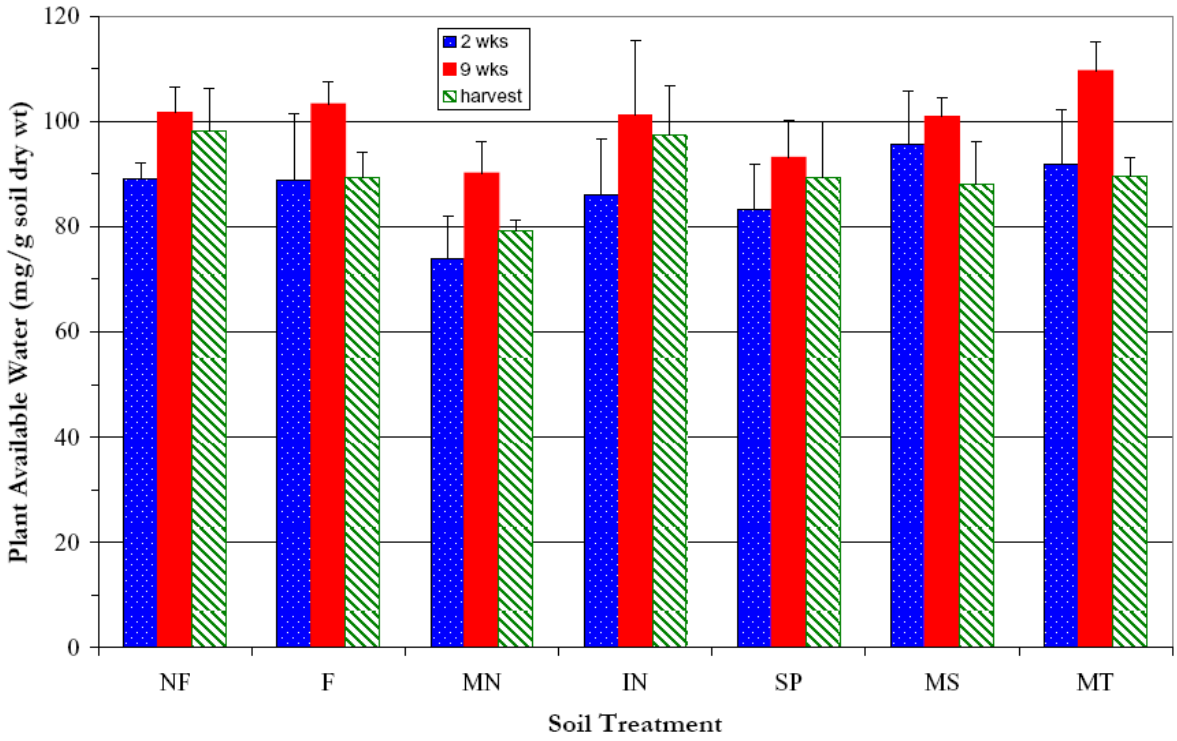


Figure 5.3-14: Plant available water for soil treatments**:** NF (unfertilized), F (fertilized), MN (manure), IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day). Colored columns indicate time from planting and are coded as follows: 2 weeks (blue textured), 9 weeks (red) and harvest (green striped). Error bars are 1 standard deviation.

similar to the results of Foley et al. (2002) who amended soil with composted or uncomposted paper mill residuals (PMR). After the first year of application, there were neither statistically significant differences between treatments, nor between the amended plots and negative control, at wilting point or with plant available water. Similar results occurred at field capacity, with the exception that the uncomposted PMR amended plots, were statistically lower than any other plot.

In the present study, the lower PAW in the MN amended soil may have been due to the differences in particle size between the manure (MN) and the composts (IN, SP, MS, MT). The smaller particles of the manure may have clogged the larger pores of the soil (Serra-Whittling et al., 1996b), whereas in the compost amended soils, this phenomenon may not have occurred to as great an extent. A similar phenomenon appears to have occurred at field capacity in the study of Foley et al. (2002), although it was not noted by the authors. However, they did mention the fine particle size of the PMR. Additionally, at wilting point, while the uncomposted PMR did not produce the statistically lowest WHC, numerically it was the smallest (with the greatest standard deviation, possibly contributing to the non-significance). In the present study, although treatment effect was not statistically significant at both field capacity and wilting point, numerically, MN had the lowest WHC at the former and next to lowest WHC at the latter.

These low values of MN apparently became manifest as statistical differences in treatments in the calculation of PAW. The trends in the present study are similar to those of Foley et al. (2002) and appear to support the clogging hypothesis. Additionally, as the effects of exogenous OM on soil structure is related to soil texture (Chapters 5.3.3.8), the similarities of the soil textures in the present study (fine, sandy loam) and in the investigation of Foley et al. (2002) (loamy sand) may have been a contributing factor in producing comparable results. Reductions in WHC (FC and WP) were also reported by Yuksel et al. (2004) after amending high clay-content soils with municipal solid waste compost, the extent of reductions being dependent upon the type of clay and application rate of amendment.

Amending soil with OM can result in increased WHC by increasing porosity, modifying pore size distribution or increasing active surface area. Increased porosity (ie, reduced bulk density) can occur by a dilution effect, while increased soil particle aggregation can also increase total porosity as well as modify pore size distribution; the higher cation exchange capacity of

OM compared to soil particles can also enhance water retention (Khaleel et al., 1981; Foley et al., 2002). These alterations in soil characteristics may affect WHC at field capacity and/or wilting point (Clapp et al., 1986), which may result in only minor modifications in PAW. The general lack of treatment effects on WHC and WAS in the present study, appears to affirm the results of Ray et al. (2006), who reported a high, positive correlation between maximum WHC and WAS, and Vepraskas et al. (1990), who reported a general positive correlation between PAW and aggregate size distribution (determined via dry sieving).

In the present study, while the change in bulk density was affected by treatment, this was not reflected in WHC, although the high sample variability in the WHC, especially at wilting point, may have masked treatment differences. Changes in soil bulk density are affected by the density of the amendment (dilution effect) and changes in porosity induced by structural rearrangement of soil particles (Czachor et al., 1995) due to aggregation effects (Oades, 1984). Bulk density is based on total porosity and is not necessarily related to pore size distribution. Therefore, changes in bulk density may not cause corresponding modifications in WHC, which is based not only on total porosity, but size of pores.

Since the WHC at both field capacity and wilting point in the present study were minimally affected by the different amendments, pores greater than approximately 30 μm may have been created. This speculation is predicated on the fact that at -0.01 MPa (field capacity) the maximum size pore water-filled is approximately 30 μm, and at -1.5 MPa (wilting point) the maximum size pore water-filled is approximately 0.20 μm (Carter et al., 1993). Similarly, Aggelides et al. (2000) concluded that amending soil with compost increased the number of larger pores, particularly approximately 60 μm in a loamy soil.

In the present study, soil was sampled at 2 and 9 weeks following planting and at harvest (approximately 3 months following planting). Effects on WHC could have been detected within this time frame since alterations in pore size distribution can occur within a short time following organic amendment application. Pagliai et al. (1981) reported that compared to an unamended control soil, amending soil modified the pore size distribution by reducing the number of very large (> 500 μm) pores, with the soil being sampled 1 and 3 months following amendment application. Additionally, in short-term studies (maximum time approximately 1 year) the effects of exogenous OM on soil WHC have been inconsistent. Although many investigators have

reported that OM has tended to increase WHC, or at a minimum has had no effect (Mbagwu, 1989; Serra-Wittling et al., 1996b; Aggelides et al., 2000; Chambers et al., 2002; Gonzalez et al., 2002), Yuksel et al. (2004) reported reduced WHC at field capacity and wilting point.

5.3.3.5 Calorific Values

Calorific values did not predict changes in soil physical properties as inferred by the original hypothesis that calorific values would be indicative of the potential for improvement of soil quality. Although the calorific values of the amendments on the day of application were different, the lack of treatments effects on soil physical properties did not confirm the hypothesis.

5.4 Field Study - Plant Response

5.4.1 Harvest Criteria

The sorghum was harvested when the grain moisture content of each plot was approximately 35%; physiological maturity for sorghum occurs when the grain moisture is approximately 25-35% (Vanderlip, 1993). This moisture level was selected somewhat arbitrarily, although grain maturity was the objective. Initially, when the grain in any plot appeared mature, the "harvest period" commenced. The grain moisture content (35.1%) of the first plot harvested (MT10) became the benchmark for the remaining plots, which were harvested when the grain moisture levels were 34.1-35.1%. Table 5.4-1 indicates each field plot, the harvest dates and grain moisture contents at harvest.

Table 5.4-1: Sorghum harvest dates and grain moisture contents

Plot Designation	Harvest Date	Grain Moisture (%)
NF1	9/19	34.1
NF2	9/19	34.3
NF3	9/19	34.3
F1	9/14*	35.0/32.9*
F2	9/8	34.9

Table 5.4-1 (continued)		
F3	9/14*	34.2/32.4*
MN1	9/19	34.8
MN2	9/14*	34.4/32.8*
MN3	9/14*	34.3/32.5*
IN1	9/19	34.1
IN2	9/8	34.8
IN3	9/19	34.6
SP6	9/14*	34.5/33.0*
SP13	9/19	34.4
SP16	9/19	34.1
MS7	9/19	34.5
MS8	9/7	34.8
MS9	9/8	34.7
MT10	8/30	35.1
MT11	9/14*	34.5/32.8*
MT12	9/8	34.2

* Harvest planned for 9/14, but due to muddy field, not harvested until 9/17. Moisture contents are from 9/13 and 9/17.

5.4.2 Yields

5.4.2.1 Grain

Grain yields are presented in Figure 5.4-1. However, birds foraged on the grain in every plot, with damages ranging from an estimated 5-20%. Bird damage is often severe when small fields of sorghum are interspersed with forests (Bennett et al., 1990); in the present study, the sorghum field was adjacent to a wooded area.

Treatments were statistically compared (on a per plot basis). Overall, treatment was not statistically significant (Pr = 0.083) and all treatments were statistically equivalent (Pr_{MIN} = 0.130). The coefficient of variation was 22.3%.

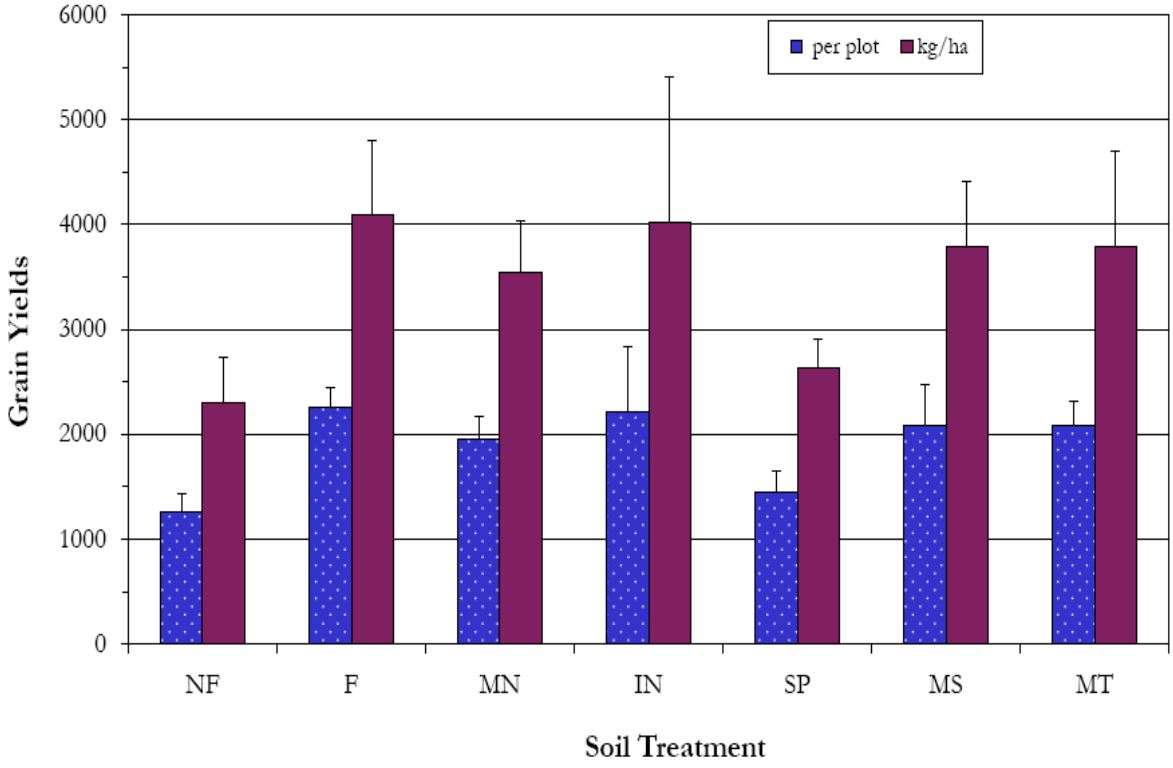


Figure 5.4-1: Sorghum grain yields normalized for 12% moisture content for each soil treatment**:** NF (unfertilized), F (fertilized), MN (manure), IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day). Blue (textured) columns are on a per plot basis and red columns are on a kg/ha basis. Blue (textured) columns with the same letter are statistically equivalent. Error bars are 1 standard deviation.

5.4.2.2 Stover

The term stover denotes the above ground plant biomass that is not harvested (ie, grain). Figure 5.4-2 shows the stover yields on a per harvested plot area basis and a per hectare basis. The statistical analysis was based on the per plot data. The effect of treatment was statistically significant (Pr = 0.007) but the effect of row was not (Pr = 0.946). The coefficient of variation was 7.8%.

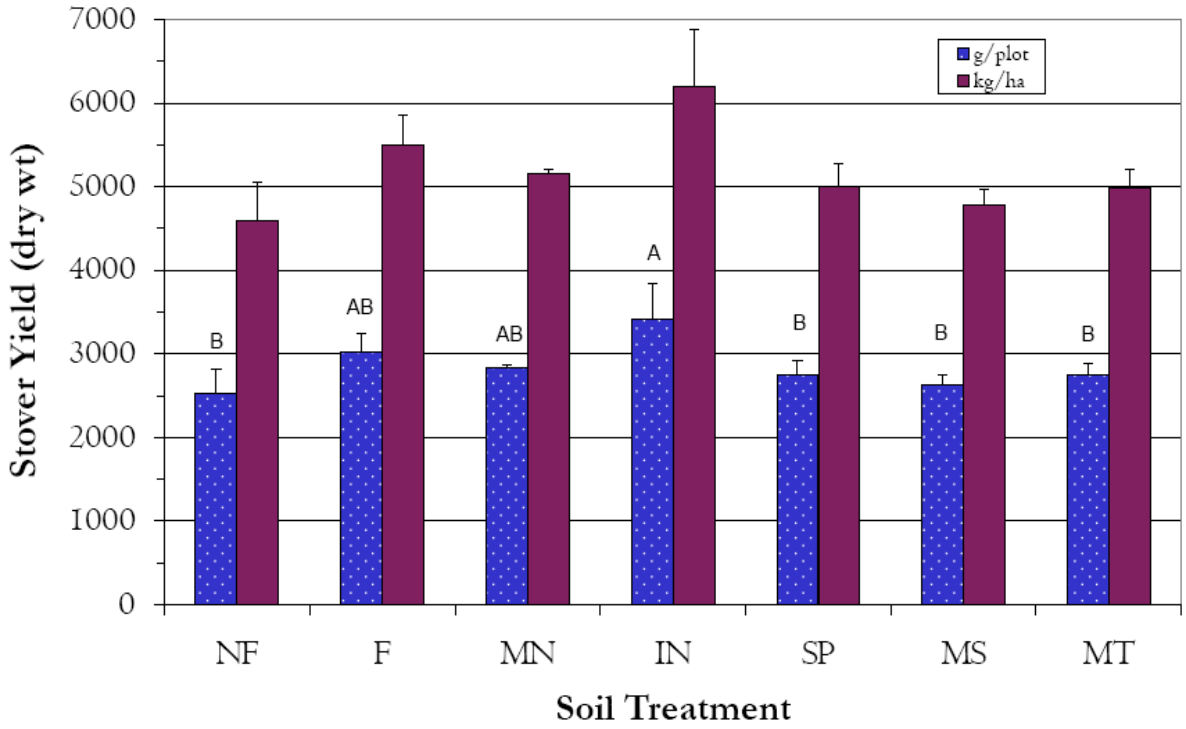


Figure 5.4-2: Sorghum stover yields for each soil treatment: NF (unfertilized), F (fertilized), MN (manure), IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day). Blue (textured) columns indicate yields per harvested plot area (g/plot) and red columns indicate yields per hectare (kg/ha). Error bars of blue columns are 1 standard deviation. Blue columns with the same letter are statistically equivalent ($\alpha = 0.05$).

The uncomposted feedstock (IN) produced the greatest yields, although they were statistically equivalent to the yields in the fertilized (F) and manured (MN) plots. As nitrogen application rates increase from insufficient to optimum, sorghum plant biomass increases (Asher et al., 1986; Kamoshita et al., 1998). However, in the present study, this trend did not occur as the unfertilized (NF) plots produced statistically equivalent yields as plots amended with MN, SP, MS and MT, as well as the fertilized control.

In the present study the uncomposted feedstock (IN) and the fertilized control (F) produced higher yields than the more composted feedstocks (SP, MS, MT). These trends are similar to those described by Cooperband et al. (2002). Averaging data from two consecutive years, the authors reported that the raw poultry litter amended soil and fertilized control

produced greater corn yields than the more composted poultry manure (1, 4 and 15 months).

Table 5.4-2 shows sorghum grain and stover yields from other investigations. The present author obtained yields by plotting nitrogen applied versus yield data and interpreting the curves. Although plant response to nutrients is dependent upon plant cultivar, crop management, edaphic factors and climate, the grain and stover yields in the present study and yields indicated in Table 5.4-2 are similar.

Table 5.4-2: Sorghum grain and stover yields from the literature

Investigator	Grain		Stover	
	Yield (kg/ha)[a]	Comments	Yield (kg/ha)	Comments
Moraghan, et al. (1984a)	5720	1981, incorporated	7970[b]	stover+chaff,[c] 1981, incorporated
Moraghan, et al. (1984b)	4910	1980, incorporated	5480[b]	stover+chaff,[c] 1980, incorporated
Nambiar, et al. (1986)	4140 2909	CSH-82 M35-1	-	-
Gakale et al. (1987)	6480 (dry wt)	in crop rotation	5840 (dry wt)	in crop rotation
Hibberd et al. (1990)	~ 5400 – 6300	range of 5 hybrids; 1st year of experiment	-	-
Hibberd et al. (1991)	2850 2400	E57 Goldfinger	-	-
Holford et al. (1997)	~ 4800 – 6700 (12% moisture)	@ 80 kg N/ha	-	-

a. unless otherwise indicated, moisture content was not indicated
b. moisture content not indicted
c. chaff = head minus grain

5.4.2.3 Harvest Index

The harvest index (HI) is a common method of assessing grain yield efficiency. The index is the ratio of grain yield (dry weight) to total plant dry weight (stover plus grain). An HI

of greater than 0.5 is considered high yield (Maiti, 1996), and is affected by total yield, since the relative amount of stover increases as grain yield decreases (Nambiar et al., 1986; Bennett et al., 1990). The indices calculated in Table 5.4-2 from data in the present study, were based on the grain yields, which were underestimates and therefore, the HI values were conservative approximations.

Table 5.4-3: Harvest Index, the ratio of grain yield to total plant dry weight

Treatment	Harvest Index	Standard Deviation
NF	0.35	0.02
F	0.42	0.03
MN	0.39	0.02
IN	0.38	0.06
SP	0.34	0.04
MS	0.41	0.04
MT	0.43	0.01

5.4.3 Nitrogen Content

Figure 5.4-3 graphically shows the nitrogen concentrations in the sorghum grain, based on 12% moisture content, including the standard deviations. Statistically, treatment effect was not significant (Pr = 0.050), nor was row (Pr = 0.633). Only treatments MT and NF were statistically different (Pr = 0.049). The coefficient of variation was 8.7%.

Figure 5.4-4 indicates the stover nitrogen concentrations based on 12% moisture, including the standard deviations. Statistically, treatment effect was significant (Pr = 0.013), but row was not (Pr_{MAX} = 0.056). The coefficient of variation was 13.5%.

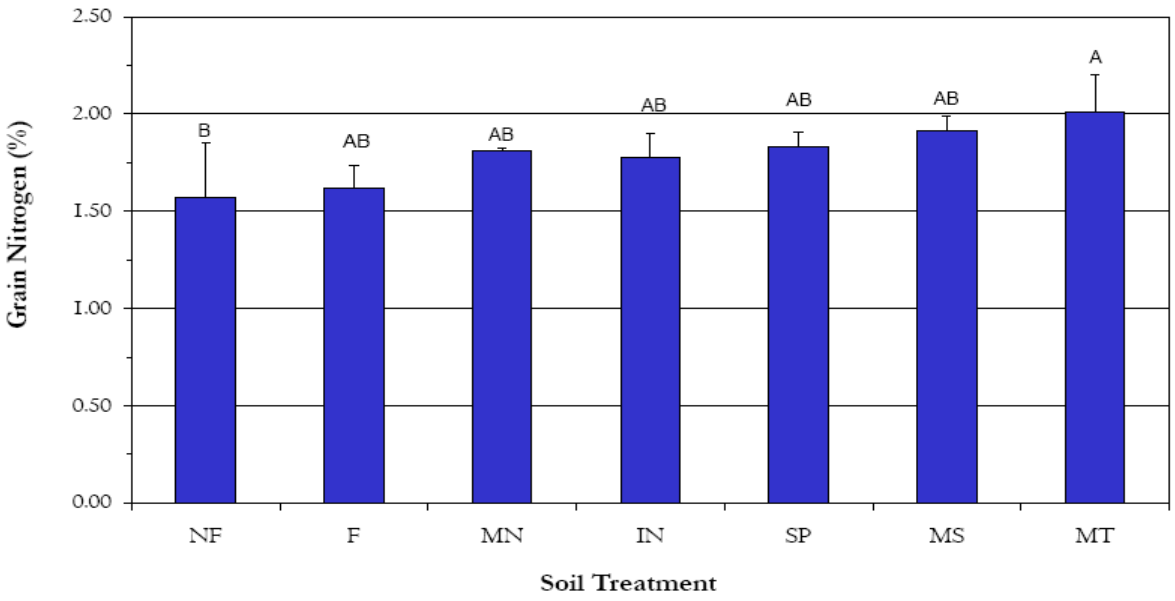


Figure 5.4-3: Grain nitrogen concentrations normalized for a grain moisture content of 12% for each soil treatment**:** NF (unfertilized), F (fertilized), MN (manure), IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day). Error bars are 1 standard deviation. Columns with the same letter are statistically equivalent (α= 0.05).

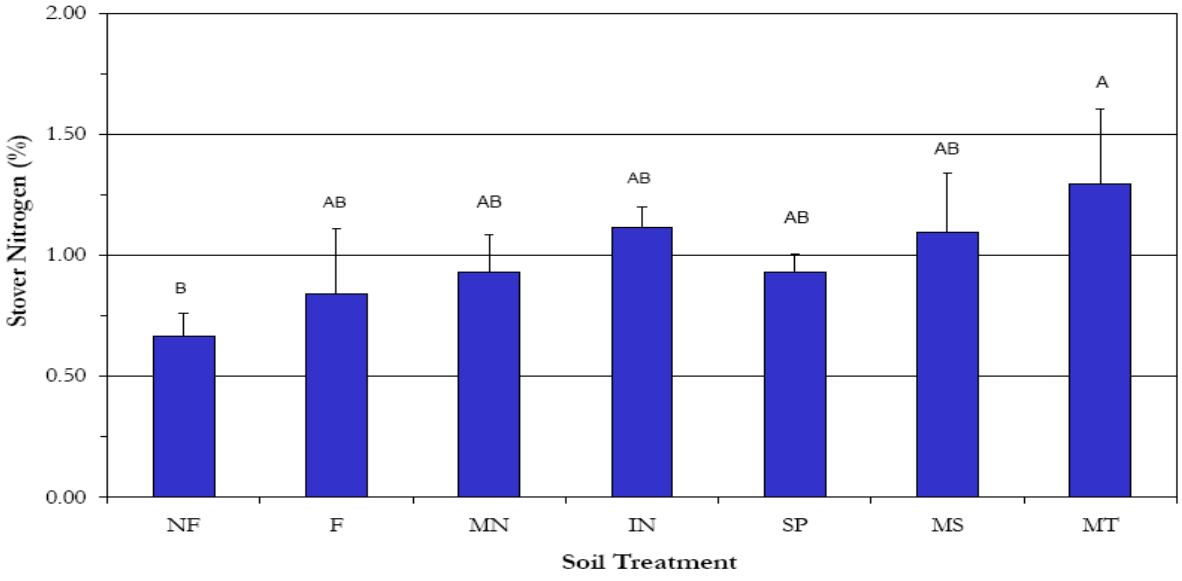


Figure 5.4-4: Stover nitrogen concentrations normalized for a biomass moisture content of 12% for each soil treatment: NF (unfertilized), F (fertilized), MN (manure), IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day). Error bars are 1 standard deviation. Columns with the same letter are statistically equivalent (α = 0.05).

The trend for nitrogen concentrations of the grain (Figure 5.4-3) and stover (Figure 5.4-4) were similar in that MT-amended soil produced the greatest uptake and unamended (NF) soils produced the lowest, with the remaining treatments falling in between. These trends were dissimilar to the stover yield trends, although in all cases NF was the treatment inducing the least response.

It was of interest to compare the grain nitrogen concentrations to other investigations to determine what levels of nitrogen could be considered adequate. The literature provided an indication of nutrient levels found in sorghum grown under a variety of conditions, with the understanding that nutrient uptake by plants is a function of edaphic, climatic and plant genotype. Comparative data from North Carolina or the Southeast was not available as sorghum is not a common crop in these areas.

Table 5.4-4 indicates nitrogen levels in sorghum grain, which were compiled from a literature review (Reuter et al., 1997) and other investigations. The numerical values were determined by the present author, from the designated sources. However, some of the data in Table 5.4-4, which were interpreted from the original journal articles, may not agree with the review by Reuter et al. (1997).

Although Table 5.4-4 provides guidance in comparing the grain nitrogen levels in the present study with the literature, direct comparisons are difficult. Given the wide variation of "adequate" levels obtained from the literature, it is uncertain what nitrogen concentrations would be defined as such in the present study. Nitrogen deficient plants do not necessarily have lower grain nitrogen concentrations (Akdeniz et al., 2006), and the conclusion from a walk-around inspection of the sorghum field about midway through the growing season was that the plants were healthy (Mueller, personal communication).

Table 5.4-4: Sorghum grain nitrogen concentrations from the literature

Nitrogen Levels (% Nitrogen)					
Marginal	**Critical**	**Adequate**	**H2O (%)**	**References**	**Notes**
1.33-1.45	-	1.62 - 1.89	14	Roy et al., 1973	-
0.93-1.02	-	1.1 - 1.2	NI*	Moraghan et al., 1984b	1981 experiment
1.2	-	1.4 - 1.5	NI	Moraghan et al., 1984b	1980 experiment, incorporated
1.13-1.22	-	1.34 – 1.39	NI	Moraghan et al., 1984a	incorporated
1.07-1.31	-	1.49 - 1.72	NI	Gakale et al., 1987	-
-	-	1.2 - 1.4	NI	Nambiar et al., 1986	genotype CSH-8R
		1.3 – 1.5	NI	Nambiar et al., 1986	genotype M35-1
-	-	1.71 - 1.86	15	Bennet et al., 1990	-
1.16-1.40	1.45	1.50 - 1.88	Moisture free	Hibberd et al., 1990	hybrids E57, P846, NK212, Goldfinger
1.39-1.49	1.7	1.81 - 2.01	NI	Hibberd et al., 1990	Goldrush hybrid
1.26-1.35	1.55	1.68 - 1.84	NI	Hibberd et al., 1991	E57 hybrid, irrigated
1.44-1.71	1.85	2.07 - 2.26	NI	Hibberd et al., 1991	Goldfinger hybrid, irrigated
1.57 (NF) – 2.01 (MT)**			12	Present Study	

* NI means not indicated

** average range for composts NF (uncomposted) and MT (103 day)

Figure 5.4-5 indicates the nitrogen accumulation in the sorghum biomass (stover plus grain) at harvest. However, the values are underestimated since the calculations included grain yields, which were low due to bird damage. The trends in Figure 5.4-5 are similar to the stover nitrogen concentrations (Figure 5.4-4); stover weight comprised approximately 2/3 of the total biomass. For net nitrogen accumulation, the overall effect of row was not statistically significant

(Pr = 0.124), nor was treatment (Pr = 0.165). The greatest difference in nitrogen accumulation occurred between treatments SP and IN, which were statistically equivalent (Pr = 0.195). The coefficient of variation was 40.3%.

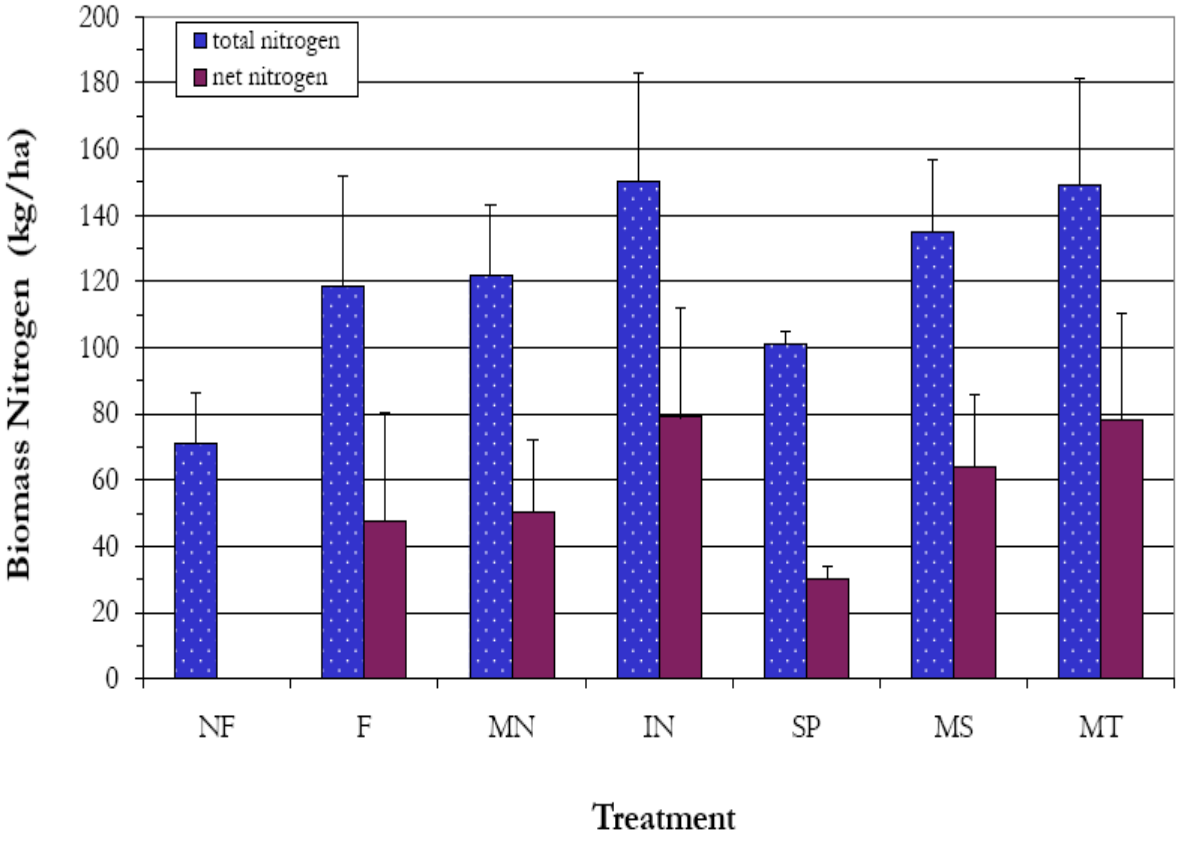


Figure 5.4-5: Nitrogen accumulation in plant biomass (stover plus grain) at harvest. Blue (textured) columns indicate total accumulation for treatments NF (unfertilized), F (fertilized), MN (manure), IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day). Red columns indicate net accumulation: total minus NF. Error bars are 1 standard deviation.

Nitrogen uptake efficiency (NUE) is the amount of accumulated nitrogen in the plant biomass divided by the amount of nitrogen applied. A high efficiency implies a high return on the nitrogen source and a reduced potential for environmental pollution. However, nitrogen uptake efficiency varies with nitrogen source, nitrogen loading rate, timing of application, plant cultivar and climatic conditions (Powell et al., 1992).

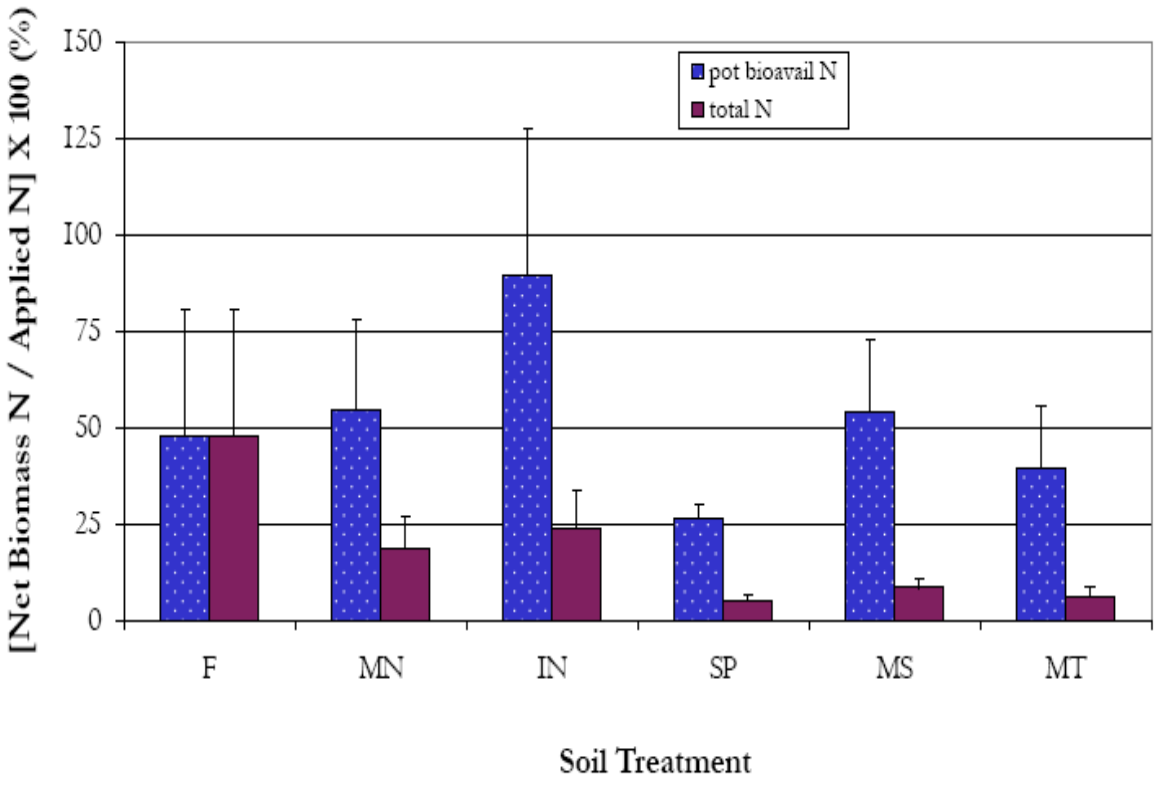


Figure 5.4-6: Net nitrogen uptake efficiency at harvest for amended plots F (fertilized), MN (manure), IN (uncomposted), SP (19 day), MS (39 day) and MT (103 day). The blue (textured) columns are based on the inorganic nitrogen extant in the amendments at the time of application plus the projected (hypothesized from the literature) amount of nitrogen that would be mineralized from the amendment during the growing season. The red columns are based on the total nitrogen applied. Nitrogen accumulation for the unfertilized control (NF) was subtracted from the nitrogen accumulation of the other treatments. Error bars are 1 standard deviation.

To calculate net nitrogen uptake efficiency (Figure 5.4-6), the nitrogen accumulation for the unfertilized control (NF) was subtracted from the nitrogen accumulation of the other treatments. The amount of nitrogen applied was either total nitrogen or "potentially available nitrogen" (Table 5.4-5). The latter was the inorganic nitrogen extant in the amendments at the time of application (Tables 4.2-4 & 4.2-5), plus the projected (hypothesized from the literature) amount of organic nitrogen that would be mineralized from the amendment during the growing season (Table 5.2-1).

Table 5.4-5: Total Nitrogen and Potentially Bioavailable Nitrogen Application Rates

Amendment	Total Nitrogen	Potential Bioavailable Nitrogen (PBN)		
	kg/ha	% Mineralized (proj) kg/ha	Inorganic N kg/ha	Total PBN kg/ha
F	101	-	101	101
MN	270	66	27	93
IN	336	79	9	88
SP	575	101	13	114
MS	765	113	5	118
MT	1287	93	105	198*

* example calculation for MT from Table 4.2-5: [(1176-9) X 0.08 + (9+96)] = 198 kg/ha

5.4.4 In Depth Discussion

5.4.4.1 General

The effects of nitrogen supply on sorghum grain and stover yields are similar, with plant stover being highly correlated with grain yield (Moraghan et al., 1984a & b; Akdeniz et al., 2006), although the relative amount of stover increases as grain yield decreases (Nambiar et al., 1986; Bennett et al., 1990). In the present study, although the grain yields were conservative estimates due to bird damage, the trends for both grain and stover yields were generally similar (Figures 5.4-1 and 5.4-2, respectively).

Plant productivity increases as soil organic matter content increases (Bauer et al., 1994) or OM loading rate increases (Akdeniz et al., 2006). This positive correlation was not reflected in the sorghum response in the present study. Compost MT provided the greatest OM loading rate and IN one of the lowest (Table 4.2-5), but stover yields were almost the reverse (Figure 5.4-2).

5.4.4.2 Nitrogen Uptake versus Yield

The organic amendments were at least as effective as inorganic fertilizer (F) enhancing nitrogen uptake (Figures 5.4-3 & 5.4-4), and stover yields (Figure 5.4-2) reflected this trend. However, total biomass nitrogen (Figure 5.4-5) indicated that SP was less effective than inorganic fertilizer in promoting nitrogen accumulation, which may have been a reflection of the lower grain yields (Figure 5.4-1). The uncomposted feedstock (IN)-amended soil produced the greatest stover yields, which were statistically equivalent (α =0.05) to the yields from the

fertilized (F) and manured (MN) plots. The unamended plots (NF) induced yields that were statistically equivalent to all other soil treatments, except IN. Grain yields for all treatments, including NF, were statistically equivalent, which may have been due to the high coefficient of variation. However, the lack of treatment effect may have also been due to higher levels of nitrogen in the soil, initially, which may have reduced plant response to added nitrogen (Holford et al., 1997).

Increasing nitrogen application rates of readily available nitrogen (eg, urea) results in increasing sorghum plant biomass and nitrogen uptake (Moraghan et al., 1984a & b; Hibberd et al., 1991; Kamoshita et al., 1998). However, grain nitrogen concentrations can be relatively high even in nitrogen deficient plants (Asher et al., 1986), since nitrogen is translocated to the grain from the vegetative parts of the plant. A greater proportion of plant nitrogen is translocated to the grain in nitrogen deficient soils compared to nitrogen sufficient soils (Asher et al., 1986; Youngquist et al., 1992), although Moraghan et al. (1984a) measured much lower stover nitrogen concentrations, compared to grain nitrogen levels, in both nitrogen-deficient and nitrogen-sufficient sorghum. In the present study, the stover nitrogen levels were much lower than the grain nitrogen levels.

The nitrogen concentrations in the grain and stover did not reflect the potentially available nitrogen applied: IN contained the least amount of available nitrogen and MT the greatest (Table 5.4-5). However, although MT induced high nitrogen uptake, IN amended soil did not produce the lowest (Figures 5.4-2 & 5.4-3). Nitrogen uptake also did not reflect the results of the mineralization study, which indicated that MN and MT had the greatest potential for producing inorganic nitrogen over the growing season (Figures 5.2-1 & 5.2-7). Although MT induced high nitrogen uptake, MN did not. Additionally, although MT contained high amounts of potentially available nitrogen and the mineralization study indicated that MT had a high potential for producing inorganic nitrogen, sorghum yields for MT did not reflect the higher availability of nitrogen. Therefore, the hypothesized nitrogen mineralization rates may have been incorrect (affecting potential nitrogen availability calculations) or the nitrogen dynamics in the incubation study and field trial were dissimilar.

Timing of nitrogen application also affects plant response since nitrogen uptake is dependent upon the timing of nitrogen availability (Moraghan et al., 1984a & b; Cowie et al.,

1986). To produce grain with adequate nitrogen concentrations, sufficient nitrogen must be supplied at planting or in split applications during the growing season (Moraghan et al., 1984a & b; Cowie et al., 1986). However, even sub-optimal nitrogen applications in a split mode, may increase grain nitrogen concentrations to a greater extent than full application at sowing (Cowie et al., 1986). The evolution of a slower release nutrient system as composting progresses (Kirchmann, 1990; Keeling et al., 1994b; Gale et al., 2006) may have some analogy to timing of nitrogen application. The gradual release of nutrients over an extended period during sorghum growth could be considered analogous to splitting nitrogen applications, and thus may have contributed to the higher concentrations of grain nitrogen (Cowie et al., 1986). Figures 5.4-3 (IN→MT) and 5.4-4 (SP→MT) show a general trend of increasing nitrogen concentrations as composting time increased. Additionally, nitrogen biomass accumulation (Figure 5.4-5) indicates a similar trend (SP→MT). Similarly, Gale et al. (2006) reported that compared to fresh dairy solids, composted dairy solids increased available (mineralized) nitrogen during incubation studies (analogous to the trend in Figure 5.2-1).

Although increasing nitrogen application rates (of available nitrogen) may increase grain nitrogen and stover yields, a nitrogen concentration effect can occur with low yields. Cowie et al. (1986) reported that grain nitrogen levels increased while the plant biomass remained constant or decreased depending upon the timing of the nitrogen application following sowing. In the present study, this phenomenon may have been exhibited with the compost amendments. The highest yields were produced in the IN-amended plots, while a trend of increasing grain nitrogen concentrations ensued as the yields decreased/remained stable with increasing composting time of the amendment.

5.4.4.3 Nitrogen Accumulation

Net biomass nitrogen accumulation (Figure 5.4-5) did not reflect the potential availability of nitrogen originally applied (Table 5.4-5). MT, which had the highest loading rate of potentially bioavailable nitrogen was statistically equivalent to IN ($Pr = 0.21$), which had the lowest loading rate, although sorghum yields for MT were slightly lower. Nitrogen accumulation tended to increase with increased composting time, although this trend was not exhibited with sorghum yields. The trend of increasing nitrogen accumulation with composting time (SP→MT) may have

been due to lower grain yields of the SP amended soil, higher potentially available nitrogen of MT (Table 5.4-5) or timing of nitrogen availability, of which the latter may be related to the effects of composting time on nutrient release.

The net nitrogen accumulation trends also did not reflect the results of the mineralization study, which indicated that MN and MT had the greatest potential for producing inorganic nitrogen (Figures 5.2-1 & 5.2-7); the nitrogen accumulation for IN was greater than either MN or MT. Therefore, it appears that the hypothesized mineralization rates were imprecise or the mineralization dynamics in the incubation study and field study were not similar, although timing of nitrogen availability may have been a contributing factor. However, although numerical differences between treatments were evident for net biomass nitrogen uptake, there were no statistical differences between treatments ($Pr_{MIN} = 0.l95$).

5.4.4.4 Nitrogen Uptake Efficiency

Based on total nitrogen applied, the inorganic fertilizer (F) produced the greatest uptake efficiency (approximately 50%) (Figure 5.4-6). The lower uptake efficiencies of the organically amended plots indicate lower nitrogen availability over a growing season, as hypothesized. The fertilizer nitrogen was comprised of ammoniacal and nitrate nitrogen which are both readily bioavailable to plants. Powell et al. (1992) reported sorghum net nitrogen uptake efficiency (NUE) for ammonium-nitrate fertilizers ranged from approximately 38% - 80% (excluding year 1988) depending upon the nitrogen loading rate, plant genotype and year. These results were based on split applications of nitrogen, which increase net NUE compared to one application at sowing (Kilcer et al., 2002), as in the present study.

Based on projected bioavailable nitrogen, net NUE of IN was the highest (approximately 90%) of the organically amended plots; SP was the lowest. Potential nitrogen availability was partially based on the availability of extant inorganic nitrogen, which was assumed to be 100%. For animal wastes, availability factors for inorganic nitrogen of 75% to 80% have been used (Beauchamp, 1986; Sims, 1987). For municipal wastewater residuals, Jacobs et al. (2002) used a factor of 100% less the amount of TAN volatilized, and Cogger et al. (2001) estimated TAN availability of 50%. In the present study, as net NUE for the fertilized control was approximately 50%, the availability factor for the inorganic nitrogen in the organic amendments may have been

closer to that value than 100%. The availability factor for inorganic nitrogen would have had the most significant impact on MT, which contained the highest initial levels of inorganic nitrogen, primarily nitrate (Table 5.4-6).

The high efficiency of IN, based on potential available nitrogen, may indicate that the hypothesized nitrogen availability (and thus nitrogen mineralization rate) and timing of availability corresponded to plant needs, although nitrogen availability may have been underestimated. Conversely, for amendments MN, SP, MS and MT, the mineralization rates may have been overestimated. However, sub-optimum timing of availability may also have contributed to lower efficiencies. Using the fertilized control as the benchmark, amendments MN, IN and MS exceeded the reference uptake efficiency, while SP and MT were less efficacious in inducing high uptake efficiency.

Composts SP and MS contained similar levels of potential available nitrogen (Table 5.4-5), although MS produced a higher net NUE. This may indicate that the slower nutrient release that evolved in the amendments with increasing duration of composting may have been more synchronous with plant needs, increasing uptake efficiency – analogous to split applications. Although MT was composted longer than MS, the uptake efficiency was lower. The slower nutrient release may have been offset by the higher inorganic loading rate of MT. Additionally, although the loading of potential available nitrogen was higher for MT than MS or SP, which promoted total uptake (Figure 5.4-5), the timing of nitrogen bioavailability for MT may have been less effective than for MS.

5.4.4.5 Available Nitrogen

Table 5.4-6 is a summary of the available nitrogen in the nitrogen mineralization (incubation) study and field trial. As previously discussed, the mineralization measured in the incubation study was lower than the hypothesized rates, except for MT, and tended to increase with composting time rather than decrease. Net bioavailable nitrogen calculated at the end of the incubation study (16 weeks), which included the initial levels of inorganic nitrogen, followed the same trend as the measured mineralization rates. However, net bioavailability measured in the incubation study was lower than the theoretical field bioavailability, except for MT. Therefore, the mineralization study accurately predicted field bioavailability for MT.

Net NUE for all treatments, based on total nitrogen applied, were lower than the theoretical field bioavailable nitrogen for all treatments except IN. Field recovery for IN was similar to the theoretical field bioavailability, which indicates that the field bioavailability calculations were accurate and availability corresponded to plant needs. The lower uptake efficiencies of the other treatments indicate that the hypothesized nitrogen mineralization rates were overestimated and/or the availability did not correspond with plant requirements. The actual mineralization rates may have been more accurately predicted by the mineralization study.

Table 5.4-6: Summary table of available nitrogen in incubation study and field trial for organic amendments (MN, IN, SP, MS, MT) and fertilizer (F)

Nitrogen, Type	Nitrogen, Source	Amendment					
		MN	**IN**	**SP**	**MS**	**MT**	**F**
Hypothesized Mineralization	% Organic N	30	25	20	15	8	-
Mineralized N, Incubation Study, 16 wks	% Organic N	9.0	4.2	2.5	5.3	10.0	-
Incubation, Net Bioavailable, 16 wks*	% Applied N	19.8	9.4	8.6	9.1	17.1	-
Theoretical Field Bioavailable, Applied**	% Applied N	34.4	26.2	19.8	15.4	15.4	100
Field N Recovery (net N Uptake Efficiency)***	% Total N Applied	18.7	23.9	5.2	8.4	6.0	47.8

* inorganic N from control soil subtracted from treatments
** calculated from Table 5.4-5
*** Figure 5.4-6

5.4.5 Summary of Conclusions

The uncomposted feedstock (IN)-amended soil produced the greatest stover yield. However, stover yield of the unamended control (NF) was statistically equivalent to all treatments, except IN. Grain yields for all treatments were statistically equivalent. Trends for nitrogen concentrations in the grain and stover were similar with MT producing the highest uptake and NF the lowest. The most composted feedstock (MT) produced a high nitrogen

uptake, which may have been due to the higher nitrogen loading or a slower nitrogen release, which is analogous to split applications. However, sorghum yields for MT did not reflect the higher nitrogen uptake.

Plant nitrogen accumulation patterns did not reflect potential available nitrogen in the amendments, nor the mineralization trends of the incubation study. Contributing factors may have been overestimation of hypothesized mineralization rates, dissimilarity of nitrogen dynamics in field trial and mineralization study and timing of nitrogen availability.

Based on total nitrogen applied, the inorganic fertilizer (F) produced the greatest nitrogen uptake efficiency. The lower uptake efficiencies of the organically amended plots indicated lower nitrogen availability, as hypothesized. Based on projected bioavailable nitrogen, net nitrogen uptake efficiency of IN was the highest of the organically amended plots; SP was the lowest. The high efficiency of IN may indicate that the hypothesized nitrogen availability (and thus nitrogen mineralization rate) and timing of availability corresponded to plant needs, although nitrogen availability may have been underestimated. For amendments MN, SP, MS and MT, the mineralization rates may have been overestimated, which may have resulted in lower amounts of amendment, and thus nitrogen applied. However, sub-optimum timing of availability may also have contributed to lower efficiencies. (Timing of nutrient application and plant requirements are discussed further in section 5.6.)

5.4.6 Short Term Field Studies

The few differences between treatments with respect to plant response are not inconsistent with other short term studies (one growing season). Brinton (1985) reported that fresh and composted manure amended soil produced yields statistically equivalent to yields from fertilized soil. Loecke et al. (2004) from the first year (2000) of their study and Brinton (1985) reported that crop yields from soils amended with either fresh or composted manures were statistically equivalent.

5.5 Calorific values

The calorific values of the amendments at the time of application did not correlate with plant response, as might be inferred from the original hypothesis which was that the calorific

values would be indicative of the beneficial potential of the amendments. While the greatest yields (Figure 5.4-1 & 5.4-2) were produced by IN amended soil, the calorific value of IN, based on OM content, was one of the lowest (Figure 5.1-21). Conversely, SP and MS had the highest calorific values at the time of field application, but the yields were low.

5.6 Best Management Practices

Agricultural best management practices (BMP) are farming methods to optimize crop growth and minimize adverse environmental effects. The methods include physically altering the terrain and managing nutrient applications to accomplish these goals. Examples of physical features are riparian buffers and contour tillage to minimize water runoff and filter sediment. Nutrient loading rates and timing of applications affect plant uptake of nutrients and potential for nutrient loss by leaching, runoff or volatilization.

Nitrogen and phosphorus are often applied in large amounts due to high requirements of plants and the relatively low concentrations existing in the soil solution (Foth, 1990). However, high concentrations of either nutrient in surface waters can cause eutrophication (excessive growth of algae and aquatic weeds causing low oxygen conditions), and drinking water supplies are limited to less than 10 mg nitrate/L. Phosphorus is relatively immobile in soil and therefore leaching into groundwater is of less concern than runoff from agricultural fields either in dissolved or particulate form. Conversely, nitrogen as nitrate is readily leached, while ammoniacal nitrogen and nitrate can be transported in surface runoff. Additionally, although phosphorus compounds are not volatile, nitrogen losses, especially due to ammonia volatilization are of concern. Consequently, as there are more pathways for potential environmental pollution for nitrogen compared to phosphorus, timing of application of nitrogen sources is more critical. Synchrony of plant available soil nutrients and crop nutrient demand is essential for optimum crop performance and environmental protection (Magdoff, 1995). However, cropping system limitations, weather and climatic conditions and field accessibility also have to be considered when managing nutrient application (NRCS, 2004).

In the present study, the amendments were applied based on the nitrogen need of sorghum. As phosphorus application was not recommended for the field trial, the additional loading of phosphorus (Table 4.2-5) may have increased the potential for phosphorus runoff. In

North Carolina, when manures or other organic byproducts are applied to agricultural fields, current best management practices dictate that phosphorus loading be considered, to minimize overloading the soil of that nutrient (NRCS, 2004).

In the present study, the nitrogen uptake efficiencies (Figure 5.4-6) varied between soil treatments. Nutrient loading, hypothesized availability of nitrogen and timing of availability were contributing factors to the variability. The high uptake efficiency of the IN amended soil (approximately 90%), based on potential available nitrogen, indicated that the three aforementioned factors were nearly optimized for a growing season, while SP amended soil produced the lowest efficiency. Therefore, it appears that the nutrient load and availability of nitrogen for IN were in concert with sorghum needs and SP was the least in synchrony (Figure 5.5-1). This is supported by the similar nitrogen accumulation trends (Figure 5.4-5).

The uptake efficiencies of MN and MS were similar to the fertilized control but would be considered less of an environmental risk since the initial nitrate loading was less than for the inorganic fertilizer, and initially plant requirements for nitrogen are relatively low (Figure 5.6-1). Conversely, MT contained the highest level of nitrate, yet had a relatively low uptake efficiency, indicating that a high initial loading of a soil mobile nutrient such as nitrate, was not in synchrony with plant needs. Consequently, the low uptake efficiency would warrant concern about potential leaching of nitrate and environmental pollution. The higher uptake efficiency of the more composted feedstock, MS, compared with the less composted, SP, indicates that the slower nutrient release transformation that occurs with increasing duration of composting may be beneficial by coordinating the nutrient availability to the plant needs, and consequently reducing adverse environmental issues. Although MT was composted for the longest time, the lower net NUE indicated that slow nutrient release may have been compromised by the initial high loading of inorganic nitrogen compared to MS. However, potential available nitrogen was based on projected nitrogen mineralization and the hypothesized rates for MN, SP, MS and MT, may have been overestimated, thus affecting apparent nitrogen uptake efficiencies. Uptake efficiencies, based on total nitrogen, which were much lower (except for the fertilized control) than the efficiencies based on potential available nitrogen do not reflect bioavailability of nitrogen and are thus less useful in assessing nutrient management optimization.

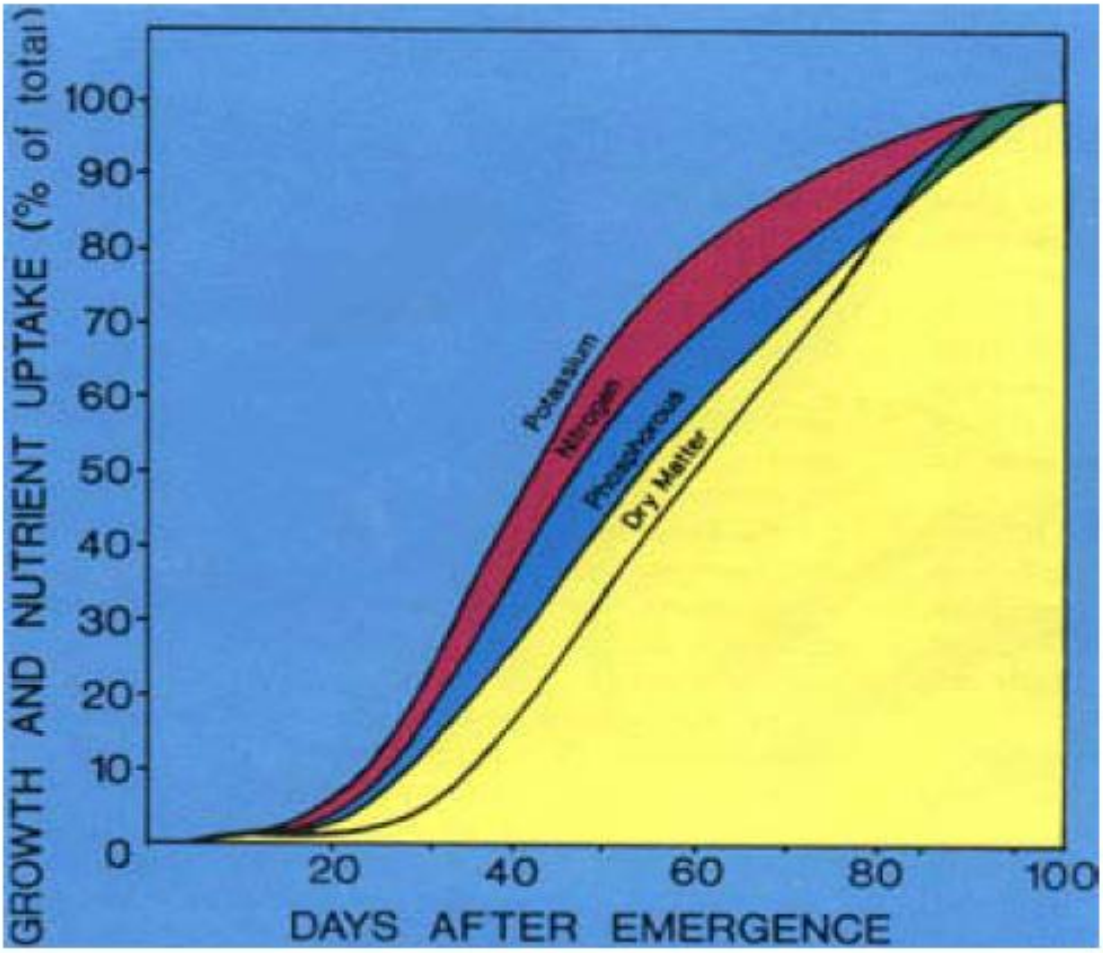


Figure 5.6-1: Growth and nutrient (nitrogen, phosphorus and potassium) uptake for sorghum. (Vanderlip, 1993)

5.7 Modeling Composting Process

Stability indices were originally going to be used as variables for relating compost characteristics to plant growth and soil characteristics. However, since soil treatment induced few significant effects in either, the original objective was not feasible.

Chapter 6
Conclusions

1. The hypothesis that 15 weeks of composting would produce a stable swine manure/wood-leaf product appeared to be confirmed based on numerical stability indices. Values for C:N ratio, nitrate-nitrogen and NH_4^+/NO_3^- ratio indicated that a stable compost had been produced, although the level of total ammoniacal nitrogen did not clearly indicate stability.

2. The hypothesis that calorific values of the compost would be indicative of the potential of the organic amendments to induce soil changes with the consequence of improved plant growth was not verified, as the calorific values were statistically invariant with respect to duration of composting. This may have been due to opposing microbial metabolic activities, the degradation of lignin and the formation of humified structures. If this was the case, then feedstocks with low lignin contents may benefit from being characterized using calorimetry.

3. The trend of decreasing nitrogen mineralization rates with increasing duration of composting was not confirmed in the incubation study. With the exception of MT, the mineralization rates in the incubation study were lower than projected. High microbial activity, due to a high nutrient loading rate, may have caused the lower measured mineralization rates by creating low oxygen conditions. Higher moisture contents may have contributed to potentially low oxygen conditions by lowering the threshold for oxygen deficit.

The hypothesized mineralization rates for the organic amendments were based upon data from the literature. Decomposition dynamics are affected by organic matter composition, soils characteristics and environmental conditions. Therefore, extrapolating results from other investigations to different specific conditions is difficult.

4. The application rates of the amendments in the field study were based upon projected nitrogen mineralization rates from previous investigations. If the mineralization dynamics in the

incubation study were similar to the soil processes in the field study, then the application rates of the amendments in the field study were suboptimal.

5. The organic amendments had a diminished effect on soil physical properties. Soil bulk density was reduced, but soil impedance and aggregate stability were unaffected; the manure (MN) tended to reduce water holding capacity. The low CEC of the soil and the interactions of calcium (extant and exogenous) and the copper and zinc (in the manure) with the inorganic and organic fractions of the soil appear to have reduced potential reactivity of the organic amendments. Additionally, the general lack of differences between treatments may have been related to the possible low amendment application rates.

6. The greatest plant nitrogen accumulation was produced in the IN and MT amended soils. Although nitrogen accumulation tended to increase with increased composting time, this trend was not exhibited with sorghum yields. Plant yields and plant nitrogen concentrations and accumulation are positively correlated with nutrient loading rates. Consequently, the diminished plant response may have been related to the possible sub-optimal loading rates of the organic amendments.

7. The high efficiency of IN, based on potential available nitrogen, may indicate that the hypothesized nitrogen availability (and thus nitrogen mineralization rate) and timing of availability corresponded to plant needs, although nitrogen mineralization rates may have been underestimated. Conversely, for amendments MN, SP and MS the mineralization rates may have been overestimated. However, for MT, the mineralization rates measured in the incubation study appeared to confirm the nitrogen bioavailability in the field study. Sub-optimal timing of nitrogen availability may also have contributed to lower efficiencies. The slower nutrient release that evolves in feedstocks during composting may be an effective means of enhancing nitrogen uptake efficiency by synchronizing nitrogen availability and plant requirements

The varied nitrogen uptake efficiencies emphasize the need for understanding nitrogen dynamics after amending soil with swine manure composts. Nitrogen mineralization potentials and timing of nitrogen availability are crucial for enhancing nitrogen uptake efficiency and minimizing adverse environmental effects.

8. Determining relationships between amendment characteristics and soil properties or plant growth was not possible since there was a general lack of significant differences between treatments.

9. Three suggestions are offered for improving the experimental design:

a. Amendment loading rate can significantly affect soil characteristics and plant growth, and as projecting nitrogen mineralization rates is risky, multiple loading rates should be included as an amendment variable.

b. It would be prudent to include different soil types to assess amendment response.

c. A multiyear field trial would be more effective in ascertaining amendment effects, as climatic conditions can dramatically change from year to year, having profound effects on soil microbial activity.

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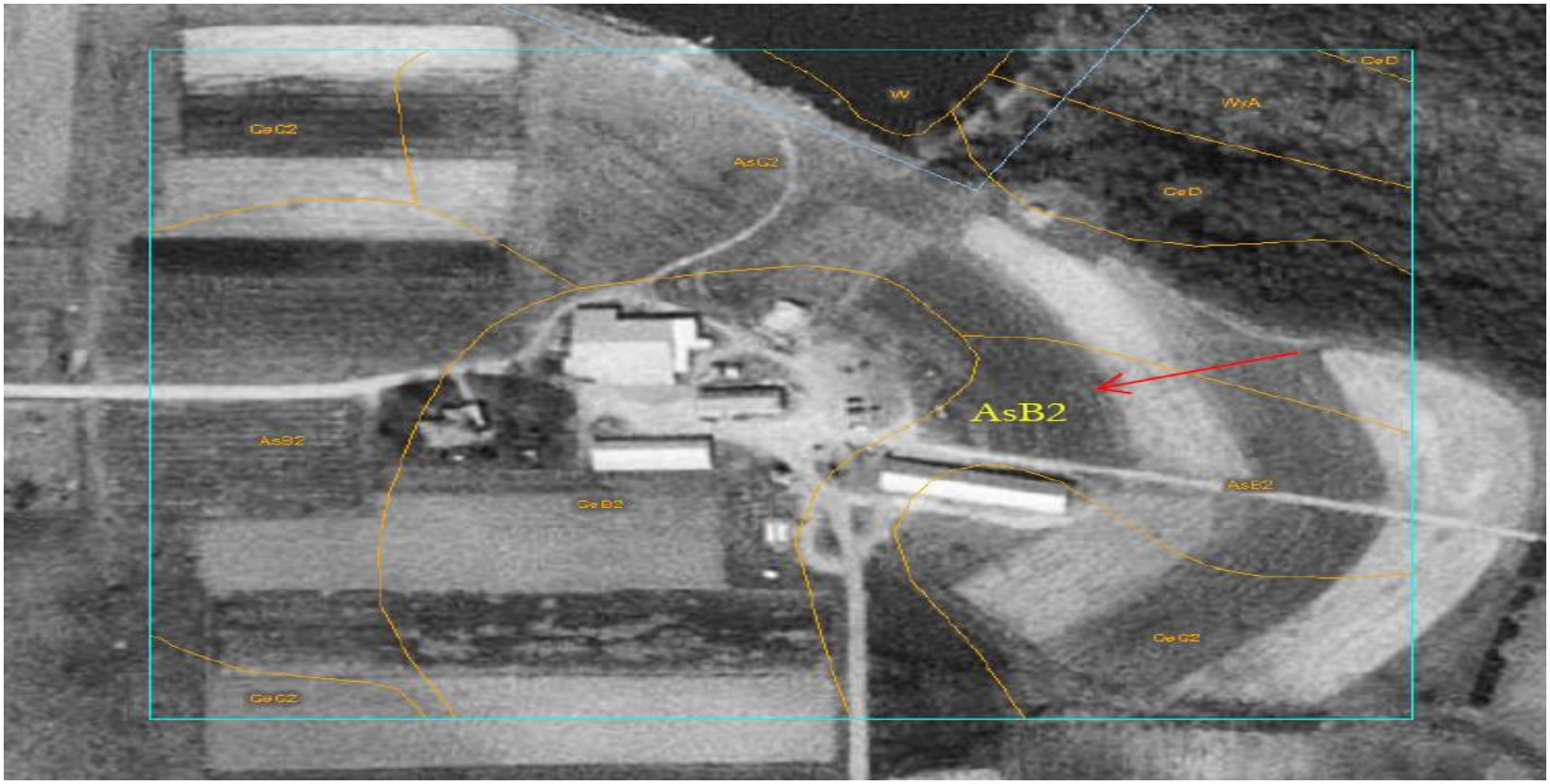
APPENDICES

Appendix A

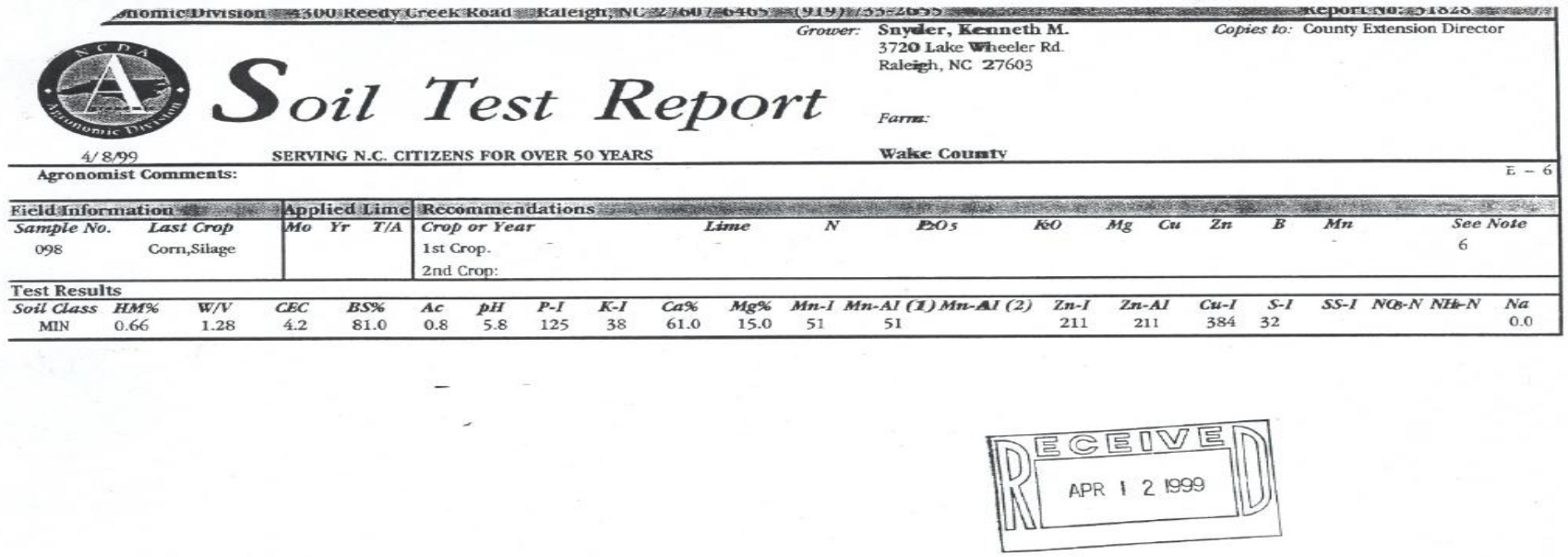
Field Location – Lake Wheeler Road Field Laboratory



The dark colored field, classified as AsB2, and indicated by the arrow was the location of the field study at the Lake Wheeler Road Field Laboratory. AsB2 designates a soil as an Appling fine, sandy loam, with 2-6% slope and moderate erosion.

Appendix B

NCDA & CS Soil Test Report



Agronomic Division 4300 Reedy Creek Road Raleigh, NC 27607 (919) 733-2655 Report No: D1828

Soil Test Report

SERVING N.C. CITIZENS FOR OVER 50 YEARS

Grower: Snyder, Kenneth M.
3720 Lake Wheeler Rd.
Raleigh, NC 27603

Copies to: County Extension Director

Farm:

Wake County

4/8/99

Agronomist Comments:

E – 6

Field Information		Applied Lime			Recommendations										
Sample No.	Last Crop	Mo	Yr	T/A	Crop or Year	Lime	N	P2O5	K2O	Mg	Cu	Zn	B	Mn	See Note
098	Corn,Silage				1st Crop.										6
					2nd Crop:										

Test Results

Soil Class	HM%	W/V	CEC	BS%	Ac	pH	P-I	K-I	Ca%	Mg%	Mn-I	Mn-AI (1)	Mn-AI (2)	Zn-I	Zn-AI	Cu-I	S-I	SS-I	NO3-N	NH4-N	Na
MIN	0.66	1.28	4.2	81.0	0.8	5.8	125	38	61.0	15.0	51	51		211	211	384	32				0.0

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The recommendations for sorghum grain were not included since at the time of soil sample submission, there was uncertainty about the crop that was going to be planted. Recommendations for sorghum nutrients were obtained from discussions with NCDA & CS and Randy Weisz (Department of Crop Science, NCSU).

Appendix C

Basis for Nitrogen Mineralization Rates

A literature review was conducted to determine the nitrogen mineralization rates of animal manures. Swine manure was of particular interest, although other animal manures were included to elucidate the effects of composting on nitrogen mineralization. Table C-1 indicates the data that was considered when projecting the mineralization rates of the swine manure/wood-leaf compost. In all of these studies, the wastes were incubated for specified times and inorganic nitrogen was determined.

Table C-1: Nitrogen Mineralization Literature Review

Organic Material	Incubation Time	% Nitrogen Mineralization		Investigators
Manures	**Days/Weeks**	**TN** [1.]	**TON** [1.]	
Swine Manure				
Swine manure	10 wk	~34		Castellanos et al., 1981
Swine manure	16 wk		31-35 [2.]	Chae et al., 1986
Swine manure	9 wk	~5 [3.]		Bernal et al., 1992
Uncomposted versus Composted Feedstocks				
Dairy manure	10 wk	~8.5/~21 [4.]		Castellanos et al., 1981
Composted dairy manure	10 wk	~6 [5.]		Castellanos et al., 1981
Chicken manure	10 wk	~48		Castellanos et al., 1981
Composted chicken manure	10 wk	~28		Castellanos et al., 1981
Broiler litter	8 wk		~38 [6.]	Tyson et al., 1993
Composted Broiler Litter	8 wk		~5 [7.]	Tyson et al., 1993"

Table C-1 (continued)				
Duration of Composting				
Fowl manure (FM) + grass clippings (GC) + coal (C) [8.]	81 d		12.7 [10]	Robertson et al., 1995
Composted FM/GC/C-2 [9.]	81 d		14.2 [10]	Robertson et al., 1995
Composted FM/GC/C-4	81 d		11.6 [10]	Robertson et al., 1995
Composted FM/GC/C-8	81 d		10.8 [10]	Robertson et al., 1995
Composted FM/GC/C-12	81 d		6.6 [10]	Robertson et al., 1995
Composted FM/GC/C-16	81 d		2.8 [10]	Robertson et al., 1995

1. TN = total nitrogen; TON = total organic nitrogen
2. range for Lester and Webster soils
3. average of fresh and aerobically treated manure
4. two different dairy manures
5. average of 3 manures
6. average of whole and sieved; Hiawassee soil
7. average of covered and uncovered; Hiawassee soil
8. initial mix
9. numbers indicate weeks of composting
10. the present author's calculations

In Table C-1, the "swine manure" section lists investigations considered when estimating the nitrogen mineralization rate for the manure. The "uncomposted versus composted feedstocks" section includes investigations indicating that composting reduced the mineralization rate, based on initial and final inorganic nitrogen concentrations. The study by Robertson et al. (1985), in the "duration of composting" section showed that nitrogen mineralization tended to decrease with increased composting time, based on periodic sampling.

In all of the investigations, except Chae et al. (1986), nitrogen mineralization calculations included subtracting initial concentrations of inorganic nitrogen from total inorganic nitrogen. Chae et al. (1986) used a leaching technique to determine the concentration of soluble inorganic nitrogen, although at the end of the study a soil analysis was also included to provide a

comprehensive inorganic nitrogen accumulation for the whole incubation study, which was 26 weeks. Consequently, the mineralization rates for less than the whole incubation period, calculated by the present author, were probably underestimates, as only the water soluble inorganic nitrogen was included.

The mineralization rates for the swine manure-based amendments, based on initial amounts of organic nitrogen, were projected as follow:

Swine manure: 30%
Initial compost mix: 25%
2 week compost: 20%
4 week compost: 15%
Mature compost: 8%

Appendix D

Mineralization Study

The amendment mixing rates were higher than the recommended agronomic rates due to the inhomogeneity of the compost. A maximum rate of 220 lb N/ac (246 kg N/ha) was recommended by the NCDA & CS for sorghum silage/forage. However, for a laboratory study, this would have necessitated using very small amounts of compost, and the less composted feedstocks were inhomogeneous, especially IN and SP. Therefore, the application rate was increased to 3000 lb N/ac (3360 kg N/ha) to reduce inhomogeneity effects. This nitrogen application rate was equivalent to 1750 mg/kg dry wt, based on a 15 cm plow depth, and a soil density of 1.28 g/cm^3 (Appendix B). Mixing rates of the amendments (Table D-1) were calculated based on amendment characteristics (Table D-2) and the aforementioned soil properties.

Table D-1: Amendment Mixing Rates and Initial Moisture Contents of the Amendment/Soil Mixtures

Soil Amendment	Amendment Weight (dry wt) per 200 g Soil (dry wt)	Calculated Moisture Content (%) of Amendment/Soil Mix
Manure (MN)	19.4 g	21.3
IN compost	21.9 g	19.3
SP compost	14.0 g	16.4
MS compost	10.3 g	16.1*
MT compost	10.6 g	16.5

* after adjusting MS moisture content to 50%

Table D-2: Nitrogen Concentrations and Moisture Contents of Organic Amendments

Soil Amendment *	Average Nitrogen Concentration (% dry wt)	Average Moisture Content (%)
Manure (MN)	1.8	71
IN compost	1.6	65
SP compost	2.5	46
MS compost	3.4	44
MT compost	3.3	52

* samples collected on day of application

The initial moisture contents of the mixtures (Table D-1) were determined with the objective of obtaining a total moisture content equivalent to 50% moisture for the amendments and 95% "container capacity" for the soil. This was a theoretical calculation to determine total water contents of the mixtures, and subsequently amounts of water that might be needed to be added to mixtures to achieve the target levels. Water was added to the SP, MS and MT amended soils. The IN and MT amendments contained sufficient water to achieve the equivalent target moisture contents without water additions.

Container capacity for the soil was determined as follows: Two hundred grams of soil (dry weight) were saturated with deionized water and allowed to drain for 24 hours. The soil was reweighed, then dried at 105° C overnight. The "container capacity" was determined to be 32 g water. A 95% container capacity (30 g/200 g dry soil) was designated as the target water holding capacity.

The following steps indicate the calculations for determining the moisture content of an amendment/soil mixture, and normalizing an amendment/soil mix to 95% soil container capacity. Manure (MN) is used as an example:

Moisture content

1) 200 g soil (dry wt) @ 5.8% moisture = 212 g compost (wet wt) = 12 g water

2) @ 71% moisture, 19.4 g manure (dry wt) = 66.9 g manure (wet wt) = 47.5 g water

3) moisture content: [(12+47.5)/(212+66.9)] X 100 = 21.3%

Normalizing amendment/soil mix to 95% soil container capacity

4) 95% container capacity = 30 g water

5) 30 -12 = 18 g water needed to achieve target WHC

6) 18 g water from manure can be "moved" to the soil to achieve 95% container capacity:

47.5 g – 18 g = 29.5 g water remain in manure

7) moisture content of manure after normalization:

[29.5/(19.4+29.5)] X 100 = ~ 60%

Appendix E

Climatological Data: Part I

Table E-1 shows monthly temperature and precipitation data for the Lake Wheeler Road Field Laboratory. Missing data can be estimated with the information from Site 317074, which is near the Lake Wheeler Road facility, at a similar latitude and longitude (Ashley Frazier, State Climate Office of NC, personal communication).

Table E-1: Temperature and Precipitation Data for Lake Wheeler Road Facility and Nearby Site 317074

STATE CLIMATE OFFICE OF NORTH CAROLINA
NC CRONOS Database
Data retrieval from LAKE - Lake Wheeler Rd Field Lab

Date	Numbr of Records Compiled	Monthly Average Daily Temperature (F)	Monthly Average Maximum Daily Temperature (F)	Monthly Average Minimum Daily Temperature (F)	Monthly Average Daily Precipitation (in)
Jun-99	30 (100%)				
Jul-99	31 (100%)	82.4	91	70.2	2.13
Aug-99	31 (100%)	78.8	90.9	69	3.25
Sep-99	30 (100%)	68.9	78.8	59.6	16.23
Oct-99	31 (100%)	59	70.3	48.4	3.29
Nov-99	30 (100%)	54.9	67.1	43.5	1
Dec-99	29 (93.5%)	44.5	55.1	33.1	2.16
Jan-00	31 (100%)	39.5	48.6	30.2	4.52
Feb-00	29 (100%)	46.5	58.4	35	1.92
Mar-00	31 (100%)	54.6	65.8	42.3	3
Apr-00	30 (100%)	57.7	68.9	46.7	2.75
May-00	1 (3.2%)	61.6	66.5	57.6	0
Jun-00	0 (0%)				

Appendix E, Continued

STATE CLIMATE OFFICE OF NORTH CAROLINA
NC CRONOS Database
Data retrieval from 317074 - Raleigh 4 SW

Date	Number of Records Compiled	Monthly AVG of Maximum Daily Temperature (F)	Monthly AVG of Minimum Daily Temperature (F)	Monthly SUM of Daily Precipitation (in)
Jun-99	30 (100%)	84.1	64.5	2.21
Jul-99	31 (100%)	90.2	70.3	2.79
Aug-99	31 (100%)	90.9	69.2	3.27
Sep-99	30 (100%)	78.9	60.3	18.47
Oct-99	31 (100%)	71.1	49.7	3.88
Nov-99	30 (100%)	68.2	44.7	1.16
Dec-99	31 (100%)	56.2	34.4	2.48
Jan-00	31 (100%)	50.6	31.1	6.13
Feb-00	29 (100%)	60	35.2	2.19
Mar-00	31 (100%)	67.9	43.4	3.42
Apr-00	30 (100%)	71.4	48.2	3.4
May-00	31 (100%)	83.1	59	0.74
Jun-00	30 (100%)	86.5	66.7	6.87

Appendix F

Climatological Data: Part II

Table F-1: Minimum and Maximum Average Monthly Temperature and Precipitation Data Since 1985: Lake Wheeler Road Facility

Air Temperature (Degrees Fahrenheit)														
VARIABLE	**STATISTIC**	**JAN**	**FEB**	**MAR**	**APR**	**MAY**	**JUN**	**JUL**	**AUG**	**SEP**	**OCT**	**NOV**	**DEC**	**Ann**
Average Temp.	**Mean**	40.1	44.5	52.5	66.3	72.6	79.6	77.9	77.1	70.6	59.9	53.3	42.5	61.4
	Highest Monthly Mean	44.2	47.3	54.6	92.4	93.4	94.5	82.4	78.8	73.2	62.2	57.2	49	61.3
	Year of Occurrence	2002	2001	2000	1999	1999	1999	1999	1999	2002	2002	2001	2001	2002
	Lowest Monthly Mean	37.1	41.4	48.5	57.7	61.6	73.8	75	74.8	68.6	58.8	48.4	36.1	59.6
	Year of Occurrence	2003	2003	2001	2000	2000	2003	2000	2000	2001	2000	2000	2000	2003

Precipitation (inches)

MONTHLY TOTAL	STATISTIC	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEP	OCT	NOV	DEC	Ann
Monthly Total	Average	2.66	2.41	4.09	1.98	1.32	2.98	4.37	5.42	5.02	3.02	1.81	2.45	37.53
	Maximum	4.83	4.29	5.78	4.54	3.1	5.83	7.89	9.7	16.23	7.58	3.43	4.07	61.39
	Year of Occurrence	2002	2003	2001	2003	2004	2001	2001	2003	1999	2002	2002	2002	2003
	Minimum	0.92	1.21	2.28	0.54	0	1.87	2.13	2.03	1.41	0	0.91	1.15	7.53
	Year of Occurrence	2004	2002	2004	2002	2000	2004	1999	2001	2001	2000	2001	2000	1998

Appendix G

Climatological Data: Part III

Table G-1: Average Monthly Temperature and Precipitation Data for Site 317074: 30 Year Average

Longitude: -78.68 degrees
Elevation: 420 feet

MEAN TEMPERATURE AND PRECIPITATION
WITH ASSOCIATED STANDAR DEVIATIONS

Analysis	**Jan**	**Feb**	**Mar**	**Apr**	**May**	**Jun**	**Jul**
30 Yr Mean-Ave Temp	41.7	44.9	52.3	60.4	68.2	75.4	79.1
30 Yr SD-Ave Temp	4.762	3.716	2.892	2.332	2.079	1.989	1.571
30 Yr Mean-Precip	4.43	3.6	4.44	2.98	3.95	4.05	4.48
30 Yr SD-Precip	1.83	1.796	1.796	1.857	1.936	2.347	2.134

	Aug	**Sep**	**Oct**	**Nov**	**Dec**	**Annual**
30 Yr Mean-Ave Temp	77.7	72.1	61.2	52.5	44.6	60.8
30 Yr SD-Ave Temp	1.935	1.854	2.791	3.01	4.032	0.902
30 Yr Mean-Precip	4.23	4.41	3.58	3.19	3.21	46.55
30 Yr SD-Precip	2.504	3.942	2.093	1.885	1.661	6.959

Appendix H

Summaries of Statistical Analyses

Note: The sections in this Appendix are numbered to correspond to those in Chapter 5

5.1 Compost Process

5.1.3 Compost Nutrients

C/N

Random Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	4	192	33.09	< 0.0001
Time	9	1686	294.12	< 0.0001
Bin (Trt) *	6	28.7		
Residual	105	66.8		

* random effect: bin nested within treatment

Contrasting Initial (i) and Final (f) Concentrations of Composts (SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
SPi – SPf	1	823	930	< 0.0001
MSi – MSf	1	352	398	< 0.0001
MTi – MTf	1	408	461	< 0.0001

Contrasting Final (f) Concentrations of Amendments (IN, MN, SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
INf – SPf	1	29.3	34.0	< 0.0001
INf – MSf	1	523.6	608.0	< 0.0001
INf – MTf	1	620.8	720.8	< 0.0001
MNf – INf	1	61.1	70.9	< 0.0001
MNf – SPf	1	22.7	26.4	< 0.0001
MNf – MSf	1	108.0	125.3	< 0.0001
MNf – MTf	1	147.0	170.6	< 0.0001
SPf – MSf	1	711.7	826.4	< 0.0001
SPf – MTf	1	885.2	1027.8	< 0.0001
MSf – MTf	1	8.3	9.6	0.003

TKN

Random Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	4	27261	24.53	< 0.0001
Time	7	13819	7.25	0.001
Bin (Trt) *	6	1716		
Residual	14	3810		

* random effect: bin nested within treatment

Contrasting Initial (i) and Final (f) Concentrations of Composts (SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
SPi – SPf	1	893	3.23	0.087
Msi – MSf	1	46.2	0.17	0.687
MTi – MTf	1	7.1	0.03	0.874

Contrasting Final (f) Concentrations of Amendments (IN, MN, SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
INf – SPf	1	1058	8.40	0.009
INf – MSf	1	5928	47.06	< 0.0001
INf – MTf	1	6391	50.73	< 0.0001
MNf – INf	1	1100	8.73	0.008
MNf – SPf	1	14.5	0.12	0.738
MNf – MSf	1	1653	13.13	0.002
MNf - MTf	1	1902	15.10	0.001
SPf – MSf	1	2472	19.63	0.0002
SPf – MTf	1	2811	22.31	0.0001
MSf - MTf	1	10.8	0.09	0.772

Total Ammoniacal Nitrogen

Random Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	4	4.746	16.28	< 0.0001
Time	7	12.111	33.78	< 0.0001
Bin (Trt) *	6	0.623		
Residual	14	0.717		

* random effect: bin nested within treatment

Contrasting Initial (i) and Final (f) Concentrations of Composts (SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
SPi – SPf	1	0.048	0.72	0.407
Msi – MSf	1	2.089	31.16	< 0.0001
MTi – MTf	1	4.751	70.88	< 0.0001

Contrasting Final (f) Concentrations of Amendments (IN, MN, SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
INf – SPf	1	0.010	0.14	0.711
INf – MSf	1	1.410	20.91	0.0002
INf – MTf	1	0.279	4.13	0.055
MNf – INf	1	3.019	44.79	< 0.0001
MNf – SPf	1	3.260	48.38	< 0.0001
MNf – MSf	1	9.550	141.69	< 0.0001
MNf - MTf	1	5.910	87.69	< 0.0001
SPf – MSf	1	2.063	30.60	< 0.0001
SPf – MTf	1	0.489	7.25	0.014
MSf - MTf	1	0.543	8.06	0.010

Nitrate

Random Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	4	19.992605	45.02	< 0.0001
Time	7	96.966979	131.54	< 0.0001
Bin (Trt) *	6	0.715299		
Residual	14	1.474317		

* random effect: bin nested within treatment

Contrasting Initial (i) and Final (f) Concentrations of Composts (SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
SPi – SPf	1	0.466	4.25	0.052
Msi – MSf	1	6.402	58.47	< 0.0001
MTi – MTf	1	65.102	594.64	< 0.0001

Contrasting Final (f) Concentrations of Amendments (IN, MN, SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
INf – SPf	1	0.023	0.19	0.669
INf – MSf	1	42.748	345.88	< 0.0001
INf – MTf	1	102.947	832.83	< 0.0001
MNf – INf	1	3.737	30.23	< 0.0001
MNf – SPf	1	31.24	31.24	< 0.0001
MNf – MSf	1	19.545	158.11	< 0.0001
MNf - MTf	1	64.458	521.45	< 0.0001
SPf – MSf	1	50.978	412.40	< 0.0001
SPf – MTf	1	124.842	1009.95	< 0.0001
MSf - MTf	1	16.268	131.61	< 0.0001

Mehlich 1 Extractable Phosphorus

Random Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	4	4.567	147.63	< 0.0001
Time	7	1.841	40.97	< 0.0001
Bin (Trt) *	6	0.058		
Residual	14	0.090		

* random effect: bin nested within treatment

Contrasting Initial (i) and Final (f) Concentrations of Composts (SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
SPi – SPf	1	0.276	37.45	< 0.0001
Msi – MSf	1	0.129	17.50	0.0005
MTi – MTf	1	0.264	35.77	< 0.0001

Contrasting Final (f) Concentrations of Amendments (IN, MN, SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
INf – SPf	1	0.160	21.74	< 0.0001
INf – MSf	1	0.147	19.97	0.0002
INf – MTf	1	0.170	22.97	0.0001
MNf – INf	1	1.332	180.48	< 0.0001
MNf – SPf	1	0.746	101.08	< 0.0001
MNf – MSf	1	0.775	105.02	< 0.0001
MNf - MTf	1	2.809	380.63	< 0.0001
SPf – MSf	1	0.0003	0.05	0.830
SPf – MTf	1	0.824	111.77	< 0.0001
MSf - MTf	1	0.791	107.22	< 0.0001

Copper

Random Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	4	51.3	6.91	0.001
Time	7	128.0	8.69	0.0003
Bin (Trt) *	6	9.0		
Residual	14	29.5		

* random effect: bin nested within treatment

Contrasting Initial (i) and Final (f) Concentrations of Composts (SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
SPi – SPf	1	39.576	20.57	0.0002
Msi – MSf	1	0.0217	0.01	0.916
MTi – MTf	1	0.297	0.15	0.698

Contrasting Final (f) Concentrations of Amendments (IN, MN, SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
INf – SPf	1	1.487	0.77	0.390
INf – MSf	1	0.012	0.01	0.938
INf – MTf	1	38.179	19.85	0.0002
MNf – INf	1	0.288	0.15	0.703
MNf – SPf	1	3.267	1.70	0.207
MNf – MSf	1	0.229	0.12	0.734
MNf - MTf	1	31.258	16.25	0.0007
SPf – MSf	1	2.208	1.15	0.297
SPf – MTf	1	68.420	35.57	< 0.0001
MSf - MTf	1	46.046	23.94	< 0.0001

Zinc

Random Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	4	3970435	6.58	0.005
Time	7	5531550	10.05	0.0002
Bin (Trt) *	6	1527359		
Residual	14	1101149		

* random effect: bin nested within treatment

Contrasting Initial (i) and Final (f) Concentrations of Composts (SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
SPi – SPf	1	643538	4.90	0.039
Msi – MSf	1	4720	0.04	0.852
MTi – MTf	1	39528	0.30	0.590

Contrasting Final (f) Concentrations of Amendments (IN, MN, SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
INf – SPf	1	167851	1.28	0.272
INf – MSf	1	893033	6.79	0.017
INf – MTf	1	4556762	34.67	< 0.0001
MNf – INf	1	585225	4.45	0.048
MNf – SPf	1	183457	1.40	0.251
MNf – MSf	1	11447	0.09	0.771
MNf - MTf	1	1681280	12.79	0.002
SPf – MSf	1	358193	2.73	0.114
SPf – MTf	1	3719363	28.30	< 0.0001
MSf - MTf	1	1769094	13.46	0.002

Phenols

Random Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	4	39300	67.32	< 0.0001
Time	7	47878	199.38	< 0.0001
Treatment*Time	1	3224	47.31	< 0.0001
Bin (Trt) *	6	1637		
Residual	59	4020		

* random effect: bin nested within treatment

Treatment*Time Effect Sliced by Treatment

Effect	DF	Sum of Squares	F Value	Pr > F
MS	3	31933	122.31	< 0.0001
MT	3	1236	4.73	0.005
SP	2	17659	101.45	< 0.0001

Contrasting Initial (i) and Final (f) Concentrations of Composts (SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
SPi – SPf	1	15715	180.58	< 0.0001
Msi – MSf	1	21372	245.58	< 0.0001
MTi – MTf	1	911	10.47	0.002

Contrasting Final (f) Concentrations of Amendments (IN, MN, SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
INf – SPf	1	4214	48.42	< 0.0001
INf – MSf	1	8306	95.44	< 0.0001
INf – MTf	1	156	1.80	0.185
MNf – INf	1	292	3.35	0.072
MNf – SPf	1	9769	112.25	< 0.0001
MNf – MSf	1	16967	194.96	< 0.0001
MNf – MTf	1	1379	15.84	0.0002
SPf – MSf	1	1681	19.32	< 0.0001
SPf – MTf	1	6366	73.14	< 0.0001
MSf – MTf	1	14755	169.54	< 0.0001

Fatty Acids

Random Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	4	21930569	2.74	0.066
Time	3	46556005	10.00	< 0.0001
Time/FA[1]	32	322183243	6.49	< 0.0001
Trt/Time/FA[2]	32	104787641	2.11	0.003
Bin (Trt) *	6	14668040		
Residual	102	158315832		

1. time*fatty acid interaction
2. treatment*time*fatty acid interaction

* random effect: bin nested within treatment

Contrasting Initial (i) and Final (f) Concentrations of Composts (SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
SPi – SPf	1	31786660	7.45	0.007
Msi – MSf	1	12298748	2.88	0.091
MTi – MTf	1	2470597	0.58	0.448

Contrasting Final (f) Concentrations of Amendments (IN, MN, SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
INf – SPf	1	1149109	0.27	0.605
INf – MSf	1	879485	0.21	0.650
INf – MTf	1	1067027	0.25	0.618
MNf – INf	1	3500584	0.82	0.366
MNf – SPf	1	11312755	2.65	0.105
MNf – MSf	1	10428298	2.44	0.120
MNf – MTf	1	11051960	2.59	0.109
SPf – MSf	1	35996	0.01	0.927
SPf – MTf	1	3041	0.00	0.979
MSf – MTf	1	18111	0.00	0.948

5.1.4 Chemical Properties

pH

Random Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	4	4.643	13.17	< 0.0001
Time	7	10.906	20.88	< 0.0001
Treatment*Time	1	0.403	5.40	0.023
Bin (Trt) *	6	0.686		
Residual	86	6.418		

* random effect: bin nested within treatment

Treatment*Time Effect Sliced by Treatment

Effect	DF	Sum of Squares	F Value	Pr > F
MS	3	0.086	0.37	0.775
MT	3	7.859	33.93	< 0.0001
SP	2	3.365	21.79	< 0.0001

Contrasting Initial (i) and Final (f) Concentrations of Composts (SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
SPi – SPf	1	1.071	13.87	0.0003
Msi – MSf	1	0.016	0.21	0.648
MTi – MTf	1	0.003	0.04	0.846

Contrasting Final (f) Concentrations of Amendments (IN, MN, SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
INf – SPf	1	3.674	47.57	< 0.0001
INf – MSf	1	1.651	21.38	< 0.0001
INf – MTf	1	3.674	47.57	< 0.0001
MNf – INf	1	0.540	6.99	.010
MNf – SPf	1	1.034	13.39	0.0004
MNf – MSf	1	0.148	1.92	0.169
MNf – MTf	1	1.034	13.39	< 0.0001
SPf – MSf	1	0.798	10.33	0.002
SPf – MTf	1	0.000	0.00	1.000
MSf – MTf	1	0.798	10.33	0.002

Electrical Conductivity

Random Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	3	3.339	25.10	< 0.0001
Time	7	7.544	43.17	< 0.0001
Treatment*Time	1	0.730	29.25	< 0.0001
Bin (Trt) *	6	0.469		
Residual	86	2.147		

* random effect: bin nested within treatment

Treatment*Time Effect Sliced by Treatment

Effect	DF	Sum of Squares	F Value	Pr > F
MS	3	1.262	14.80	< 0.0001
MT	3	3.586	42.03	< 0.0001
SP	2	3.425	60.22	< 0.0001

Contrasting Initial (i) and Final (f) Concentrations of Composts (SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
SPi – SPf	1	2.796	98.31	< 0.0001
Msi – MSf	1	0.760	26.72	< 0.0001
MTi – MTf	1	0.891	31.31	< 0.0001

Contrasting Final (f) Concentrations of Amendments (IN, MN, SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
INf – SPf	1	0.602	21.18	< 0.0001
INf – MSf	1	1.174	41.29	< 0.0001
INf – MTf	1	2.797	98.36	< 0.0001
MNf – INf	1	0.009	0.32	0.571
MNf – SPf	1	0.434	15.25	0.0002
MNf – MSf	1	0.933	32.82	< 0.0001
MNf – MTf	1	2.418	85.01	< 0.0001
SPf – MSf	1	0.189	6.65	0.012
SPf – MTf	1	1.607	56.51	< 0.0001
MSf – MTf	1	0.693	24.38	< 0.0001

5.1.5 Calorimetry

Calorific Values

Random Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	4	610149	0.86	0.510
Time	8	2335545	1.88	0.118
Bin (Trt) *	6	1176712		
Residual	21	3260107		

* random effect: bin nested within treatment

Treatment*Time Effect Sliced by Treatment

Effect	DF	Sum of Squares	F Value	Pr > F
MS	3	566795	1.15	0.347
MT	3	572806	1.16	0.342
SP	2	1195944	3.64	0.040

Contrasting Initial (i) and Final (f) Concentrations of Composts (SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
SPi – SPf	1	1053366	6.41	0.018
MSi – MSf	1	494788	3.01	0.094
MTi – MTf	1	356241	2.17	0.153

Contrasting Final (f) Concentrations of Amendments (IN, MN, SP, MS and MT)

Contrast	DF	Sum of Squares	F Value	Pr > F
INf – SPf	1	855038	5.20	0.031
INf – MSf	1	998784	6.08	0.020
INf – MTf	1	5046	0.03	0.862
MNf – INf	1	4907	0.03	0.864
MNf – SPf	1	1120581	6.82	0.015
MNf – MSf	1	1296051	7.89	0.009
MNf – MTf	1	35	0.00	0.986
SPf – MSf	1	5582	0.03	0.855
SPf – MTf	1	991454	6.03	0.021
MSf – MTf	1	1145814	6.97	0.014

5.2 Nitrogen Mineralization Study

Net Inorganic Nitrogen
Random Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment (Trt)	4	286869	95.97	< 0.0001
Time	8	393513	21.65	< 0.0001
Rep(Trt)*	10	7473		
Residuals	112			

* sample replicate (Rep, random effect) nested within treatment (Trt)

Comparison of Treatments at 16 weeks Using Tukey's (HSD) Multicomparison Test

Treatment	Mean NIN (mg/kg dry wt)[1]	Tukey Grouping[2,3]
MN	346.2	A
MT	300.2	A
IN	164.2	B
MS	159.9	B
SP	149.6	B

1. NIN = net inorganic nitrogen
2. rows with the same letter are statistically equivalent
3. least significant difference = 53.9

Percent Mineralized Nitrogen

Random Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment (Trt)	4	505	58.85	< 0.0001
Time	7	824	23.85	< 0.0001
Rep(Trt)*	10	22		
Residuals	98	483		

* sample replicate (Rep, random effect) nested within treatment (Trt)

Comparison of Treatments at 16 weeks Using Tukey's (HSD) Multicomparison Test

Treatment	Mean (% TON)[1]	Tukey Grouping[2,3]
MT	10.04	A
MN	8.98	A
MS	5.30	B
IN	4.18	BC
SP	2.46	C

1. TON = total organic nitrogen
2. rows with the same letter are statistically equivalent
3. least significant difference = 2.48

C:N

Random Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment (Trt)	5	0.421	36.92	< 0.0001
Time	1	1.071	537.19	< 0.0001
Trt*Time	5	0.556	55.78	< 0.0001
Rep(Trt)*	12	0.027		
Residuals	12	0.023		

* sample replicate (Rep, random effect) nested within treatment (Trt)

Comparisons of Initial and Final C:N Values

Treatment Contrast	DF	Sum of Square	F Value	Pr > F
S	1	0.0006	0.29	0.592
MN	1	0.781	365.52	< 0.0001
IN	1	0.590	276.15	< 0.0001
SP	1	0.12	57.80	< 0.0001
MS	1	0.084	39.44	< 0.0001
MT	1	0.045	21.69	< 0.0001

Total Nitrogen

Random Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment (Trt)	5	7.281	118.67	< 0.0001
Time	1	0.0006	0.04	0.834
Trt*Time	5	0.046	0.63	0.680
Rep(Trt)*	12	0.147		
Residuals	12	0.176		

* sample replicate (Rep, random effect) nested within treatment (Trt)

Comparisons of Initial and Final Total Nitrogen Values

Treatment Contrast	DF	Sum of Square	F Value	Pr > F
S	1	0.004	0.29	0.592
MN	1	0.003	0.21	0.654
IN	1	0.002	0.13	0.720
SP	1	0.005	0.38	0.544
MS	1	0.001	0.08	0.784
MT	1	0.032	2.40	0.135

5.3 Soil Properties
5.3.2 Soil Nutrients

Total Kjeldahl Nitrogen
Repeated Measures of Analysis

Between Subject Effects				
Source	DF	Sum of Squares	F Value	Pr > F**
Treatment	6	1735792	4.56	0.015
Row	2	362565	2.86	0.100
Residuals	11	697943		
Within Subject (Treatment) Effects				
Time	3	317861	21.56	< 0.0001
Time*Treatment	18	187042	2.11	0.030
Time*Row	6	29074	0.99	0.451
Residuals	33	162179		

** for within subject effects, probability was adjusted using Huynh-Feldt correction

Contrasting 6 month to Other Times (3, 9 and 12 months)

Contrast	DF	Sum of Squares	F Value	Pr > F
6 months vs. 3 months	1	312380	50.24	< 0.0001
6 months vs. 9 months	1	594737	54.10	< 0.0001
6 months vs. 12 months	1	175068	12.96	0.004

Nitrate
Repeated Measures of Analysis

Between Subject Effects				
Source	DF	Sum of Squares	F Value	Pr > F
Treatment	6	60	2.96	0.057
Row	2	20	2.93	0.095
Residuals	11	37		
Within Subject (Treatment) Effects				
Time	3	63	22.14	< 0.0001
Time*Treatment	18	26	1.53	0.141
Time*Row	6	11	2.07	0.084
Residuals	33	31		

Mehlich 1 Extractable Phosphorus

Repeated Measures of Analysis

Between Subject Effects				
Source	DF	Sum of Squares	F Value	Pr > F**
Treatment	6	609315	58.83	< 0.0001
Row	2	13769	3.99	0.050
Residuals	11	18990		
Within Subject (Treatment) Effects				
Time	3	68616	35.38	< 0.0001
Time*Treatment	18	28980	2.49	0.011
Time*Row	6	4802	1.24	0.313
Residuals	33	21332		

** for within subject effects, probability was adjusted using Huynh-Feldt correction

Contrasting 3 month to Other Times (6, 9 and 12 months)

Contrast	DF	Sum of Squares	F Value	Pr > F
3 months vs. 6 months	1	636	1.15	0.306
3 months vs. 9 months	1	51090	67.76	< 0.0001
3 months vs. 12 months	1	71847	41.43	< 0.0001

Contrasting 12 month to Other Times (3, 6 and 9 months)

Contrast	DF	Sum of Squares	F Value	Pr > F
12 months vs. 3 months	1	71847	41.43	< 0.0001
12 months vs. 6 months	1	86001	156.94	< 0.0001
12 months vs. 9 months	1	1765	0.66	0.432

Calcium

Repeated Measures of Analysis

Between Subject Effects				
Source	DF	Sum of Squares	F Value	Pr > F**
Treatment	6	2320752	12.96	0.0002
Row	2	38242	0.64	0.545
Residuals	11	328202		
Within Subject (Treatment) Effects				
Time	3	1144559	47.6	< 0.0001
Time*Treatment	18	452483	3.14	0.002
Time*Row	6	82527	1.72	0.148
Residuals	33	264502		

** for within subject effects, probability was adjusted using Huynh-Feldt correction

Contrasting 3 month to Other Times (6, 9 and 12 months)

Contrast	DF	Sum of Squares	F Value	Pr > F
3 months vs. 6 months	1	169134	17.25	0.002
3 months vs. 9 months	1	1326172	89.01	< 0.0001
3 months vs. 12 months	1	1708294	82.15	< 0.0001

Contrasting 12 month to Other Times (3, 6 and 9 months)

Contrast	DF	Sum of Squares	F Value	Pr > F
12 months vs. 3 months	1	1708294	82.15	< 0.0001
12 months vs. 6 months	1	802383	78.6	< 0.0001
12 months vs. 9 months	1	24156	1.25	0.278

Magnesium

Repeated Measures of Analysis

Between Subject Effects				
Source	DF	Sum of Squares	F Value	Pr > F
Treatment	6	74785	2.03	0.146
Row	2	26113	2.13	0.166
Residuals	11	67551		
Within Subject (Treatment) Effects				
Time	3	87965	24.94	< 0.0001
Time*Treatment	18	32480	1.53	0.140
Time*Row	6	17774	2.52	0.041
Residuals	33	38802		

** for within subject effects, probability was adjusted using Huynh-Feldt correction

Contrasting 3 month to Other Times (6, 9 and 12 months)

Contrast	DF	Sum of Squares	F Value	Pr > F
3 months vs. 6 months	1	22747	16.98	0.002
3 months vs. 9 months	1	10422	11.63	0.006
3 months vs. 12 months	1	162311	51.91	< 0.0001

Contrasting 12 month to Other Times (3, 6 and 9 months)

Contrast	DF	Sum of Squares	F Value	Pr > F
12 months vs. 3 months	1	162311	51.91	< 0.0001
12 months vs. 6 months	1	63533	18.31	0.001
12 months vs. 9 months	1	90473	24.97	0.0004

5.3.3 Physical Properties

Wet Aggregate Stability

Repeated Measures of Analysis

Between Subject Effects				
Source	DF	Sum of Squares	F Value	Pr > F
Treatment	6	0.037	1.17	0.384
Row	2	0.068	6.50	0.012
Residuals	12	0.063		
Within Subject (Treatment) Effects				
Time	5	0.183	26.24	< 0.0001
Time*Treatment	30	0.054	1.28	0.207
Time*Row	10	0.011	0.78	0.648
Residuals	60	0.084		

Contrasting Initial Time (5 days) to other Times (2 months, harvest, 6, 9 and 12 months)

Contrast	DF	Sum of Squares	F Value	Pr > F
5 days vs. 2 months	1	0.030	6.18	0.029
5 days vs. harvest	1	0.030	13.39	0.003
5 days vs. 6 months	1	0.007	1.87	0.197
5 days vs. 9 months	1	0.005	0.95	0.350
5 days vs. 12 months	1	0.131	34.45	< 0.0001

Contrasting Final Time (12 months) to other Times (5 days, 2 months, harvest, 6 and 9 months)

Contrast	DF	Sum of Squares	F Value	Pr > F
12 months vs. 5 days	1	0.131	34.45	< 0.0001
12 months vs. 2 months	1	0.035	19.95	0.0008
12 months vs. harvest	1	0.287	243.47	< 0.0001
12 months vs. 6 months	1	0.199	281.44	< 0.0001
12 months vs. 9 months	1	0.083	33.71	< 0.0001

Bulk Density

Repeated Measures of Analysis

Between Subject Effects				
Source	DF	Sum of Squares	F Value	Pr > F**
Treatment	6	0.2056	6.48	0.003
Row	2	0.0035	0.33	0.725
Residuals	12	0.0634		
Within Subject (Treatment) Effects				
Time	3	0.3329	62.02	< 0.0001
Time*Treatment	18	0.0308	0.96	0.522
Time*Row	6	0.0140	1.31	0.279
Residuals	36	0.0644		

** for within subject effects, probability was adjusted using Huynh-Feldt correction

Individual contrasts comparing initial time (1 week) to other times (2 months, harvest and 12 months)

Contrast	DF	Sum of Squares	F Value	Pr > F
1 wk vs. 2 months	1	0.406	179.44	< 0.0001
1 wk vs. harvest	1	0.573	117.72	< 0.0001
1 wk vs. 12months	1	0.174	91.93	< 0.0001

Repeated Measures of Analysis – Excluding Initial Sampling Time

Between Subject Effects				
Source	DF	Sum of Squares	F Value	Pr > F**
Treatment	6	0.1840	6.49	0.003
Row	2	0.0066	0.70	0.515
Residuals	12	0.0567		
Within Subject (Treatment) Effects				
Time	2	0.0596	14.36	< 0.0001
Time*Treatment	12	0.0179	0.72	0.721
Time*Row	4	0.0104	1.25	0.317
Residuals	24	0.0498		

** for within subject effects, probability was adjusted using Huynh-Feldt correction

Penetrometer Resistance

Repeated Measures of Analysis

Between Subject Effects				
Source	DF	Sum of Squares	F Value	Pr > F**
Treatment	6	123706	0.76	0.617
Row	2	39548	0.73	0.504
Residuals	12	327244		
Within Subject (Treatment) Effects				
Time	3	1445096	46.58	< 0.0001
Time*Treatment	18	122940	0.66	0.825
Time*Row	6	45848	0.74	0.622
Residuals	36	372300		

** for within subject effects, probability was adjusted using Huynh-Feldt correction

Individual contrasts comparing initial time (5 days) to other times (11, 12 and 17 weeks)

Contrast	DF	Sum of Squares	F Value	Pr > F
5 days vs. 11 wks	1	1917550	49.62	< 0.0001
5 days vs. 12 wks	1	1460713	59.18	< 0.0001
5 days vs. 17 wks	1	2267964	73.53	< 0.0001

Repeated Measures of Analysis – Excluding Initial Sampling Time

Between Subject Effects				
Source	DF	Sum of Squares	F Value	Pr > F
Treatment	6	113580	1.54	0.248
Row	2	30686	1.25	0.323
Residuals	12	327244		
Within Subject (Treatment) Effects				
Time	2	44719	4.48	0.022
Time*Treatment	12	57864	0.97	0.504
Time*Row	4	22505	1.13	0.367
Residuals	24	119723		

Water Holding Capacity

Split Plot Model

Field Capacity

Source	DF	Sum of Squares	F Value	Pr > F
Treatment (Trt)	6	10777	14.53	< 0.0001
Time	2	3362	13.60	< 0.0001
Trt*Time	12	2089	1.41	0.167
Row	2	5709	23.10	< 0.0001
Trt*Row	12	8933	6.02	< 0.0001
Tests of Hypothesis Using Trt*Row as Error Term				
Treatment	6	10777	2.41	0.092
Row	2	5709	3.83	0.052

Treatment Averages

Treatment	LS Mean
MS	145.9
MT	145.6
IN	138.2
NF	137.5
SP	135.1
F	133.0
MN	122.0

Treatment Comparisons Using Tukey-Kramer Adjustment for Multicomparisons (Pr > F)

	F	IN	MN	MS	MT	NF	SP
F		0.992	0.745	0.609	0.637	0.995	1.000
IN			0.395	0.940	0.951	1.000	1.000
MN				0.078	0.085	0.413	0.593
MS					1.000	0.906	0.759
MT						0.922	0.785
NF							1.000

Wilting Point

Source	DF	Sum of Squares	F Value	Pr > F
Treatment (Trt)	6	3084	81.37	< 0.0001
Time	2	214	16.93	< 0.0001
Trt*Time	12	266	3.51	0.0002
Row	2	3202	253.43	< 0.0001
Trt*Row	12	5175	68.27	< 0.0001
Tests of Hypothesis Using Trt*Row as Error Term				
Treatment	6	3084	1.19	0.373
Row	2	3202	3.71	0.056

Treatment*Time Effect Sliced by Time

Time	DF	Sum of Squares	F Value	Pr > F
June	6	1922	50.71	< 0.0001
August	6	588	15.52	< 0.0001
Harvest	6	930	24.54	< 0.0001

Treatment Averages

Treatment	LS Mean
MS	51.3
MT	50.0
SP	46.4
IN	43.4
NF	41.2
MN	40.8
F	39.2

Treatment Comparisons Using Tukey-Kramer Adjustment for Multicomparisons (Pr > F)

	F	IN	MN	MS	MT	NF	SP
F		0.989	1.000	0.469	0.571	1.000	0.879
IN			1.000	0.844	0.918	1.000	0.998
MN				0.640	0.744	1.000	0.963
MS					1.000	0.668	0.981
MT						0.771	0.996
NF							0.972

Plant Available Water (PAW)

Source	DF	Sum of Squares	F Value	Pr > F
Treatment (Trt)	6	1739	3.52	0.010
Time	2	1955	11.87	0.0002
Trt*Time	12	619	0.63	0.802
Row	2	85	0.51	0.604
Trt*Row	12	347	0.35	0.970
Tests of Hypothesis Using Trt*Row as Error Term				
Treatment	6	1739	10.01	0.0004
Row	2	85	1.46	0.271

Treatment Averages

Treatment	LS Mean
MT	97.1
NF	96.3
IN	94.9
MS	94.9
F	93.8
SP	88.5
MN	81.1

Treatment Comparisons Using Tukey-Kramer Adjustment for Multicomparisons (Pr > F)

	F	IN	MN	MS	MT	NF	SP
F		0.999	0.0042	.09995	.0849	.0953	0.419
IN			0.002	1.000	0.975	0.998	0.234
MN				0.002	0.001	0.001	0.127
MS					0.970	0.997	0.245
MT						0.9999	0.063
NF							0.105

5.4 Plant Response
5.4.2 Yields

Grain Yields

Fixed Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	6	2705997	2.53	0.083
Row	2	370292	1.04	0.383
Residuals	12	2135188		

Treatment Comparisons Using Tukey-Kramer Adjustment for Multicomparisons (Pr > F)

	IN	MN	MS	MT	NF	SP
F	1.000	0.972	.0.999	0.999	0.130	0.297
IN		0.985	1.000	1.000	0.155	0.343
MN			1.000	1.000	0.459	0.761
MS				1.000	0.276	0.541
MT					0.280	0.548
NF						0.998

Stover Yields

Fixed Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	6	1550505	5.30	0.007
Row	2	5414	0.06	0.946
Residuals	12	584962		

Treatment Comparisons Using Tukey-Kramer Adjustment for Multicomparisons (Pr > F)

	IN	MN	MS	MT	NF	SP
F	0.384	0.939	0.371	0.715	0.169	0.737
IN		0.085	0.013	0.036	0.005	0.039
MN			0.897	0.998	0.619	0.999
MS				0.994	0.997	0.992
MT					0.884	1.000
NF						0.869

5.4.3 Nitrogen Content

Grain Nitrogen Concentrations

Fixed Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	6	0.434	2.99	0.050
Row	2	0.021	0.43	0.633
Residuals	12	1.028		

Treatment Comparisons Using Tukey-Kramer Adjustment for Multicomparisons (Pr > F)

	IN	MN	MS	MT	NF	SP
F	0.874	0.731	0.288	0.094	1.000	0.660
IN		1.000	0.899	0.519	0.705	1.000
MN			0.973	0.689	0.535	1.000
MS				0.987	0.175	0.988
MT					0.049	0.759
NF						0.466

Stover Nitrogen Concentrations

Fixed Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	6	0.756	4.46	0.013
Row	2	0.209	3.69	0.056
Residuals	12	1.303		

Treatment Comparisons Using Tukey-Kramer Adjustment for Multicomparisons (Pr > F)

	IN	MN	MS	MT	NF	SP
F	0.465	0.993	0.532	0.066	0.866	0.991
IN		0.823	1.000	0.823	0.079	0.834
MN			0.876	0.187	0.518	1.000
MS				0.763	0.095	0.885
MT					0.009	0.193
NF						0.504

Net Nitrogen Accumulation`

Fixed Effects Model

Source	DF	Sum of Squares	F Value	Pr > F
Treatment	5	5482	1.99	0.165
Row	2	2854	2.59	0.124
Residuals	10	2135188		

Treatment Comparisons Using Tukey-Kramer Adjustment for Multicomparisons (Pr > F)

	IN	MN	MS	MT	SP
F	0.599	1.000	0.949	0.624	0.930
IN		0.681	0.966	1.000	0.195
MN			0.977	0.705	0.881
MS				0.973	0.515
MT					0.207