Changes in Soil Nitrogen Following Biosolids Application to Loblolly Pine (*Pinus Taeda* L.) Forest in the Virginia Piedmont

Eduardo C. Arellano Ogaz

Dissertation submitted to the faculty of the Virginia Polytechnic Institute and State University in partial fulfillment of the requirements for the degree of

Doctor of Philosophy

In

Forestry

Dr. Thomas R. Fox, Chair

Dr. W. Michael Aust

Dr. James A. Burger

Dr. W. Lee Daniels

Dr. Gregory K. Evanylo

January, 12, 2009

Blacksburg, VA

Keywords: Loblolly pine (*Pinus Taeda* L.), biosolids, Nitrogen mineralization, Nitrogen leaching, forest fertilization

Changes in Soil Nitrogen Following Biosolids Application to Loblolly Pine (*Pinus Taeda* L.) Forest in the Virginia Piedmont

Eduardo C. Arellano Ogaz

ABSTRACT

Application of biosolids as an alternative source of Nitrogen (N) is becoming a common silviculture practices on loblolly pine forest. However, little is known about how biosolids type, application rate, and timing affect forest floor and soil N availability in pine plantations. The objectives of this study were to determine the effect of different types, rates, and season of application of biosolids on forest floor and soil N. The study was established in a 17-year-old loblolly pine plantation in Amelia County, VA. Anaerobically digested (AD225), lime stabilized (LS225), and pelletized (Pellet225) biosolids and a conventional inorganic urea plus diammonium phosphate fertilizer (U+DAP225) were surface applied at a rate of 225 kg ha^{-1} based on Plant Available Nitrogen (PAN) between March 5th and 10th, 2006. Anaerobically digested biosolids were also surface applied at the rates of 900 kg PAN ha^{-1} and 1800 kg PAN ha^{-1} (AD900 and AD1800). Anaerobically digested biosolids at the rate of 900 kg PAN ha^{-1} were also applied on November 5th, 2005 (AD900F).

Surface application of different type of biosolids in a loblolly pine plantation increased soil N availability and mineralization when biosolids were applied at the permitted rate of 225 kg PAN ha^{-1}. Surface soil NH_4-N and NO_3-N availability and N mineralization was significantly different among biosolids type over time. N release from different type of biosolids depends on the initial inorganic N content, and N mineralization in biosolids. The average soil N availability and mineralization was significantly greater in the Pellet225 treatments than in all the other treatments. Soil N availability decreased in winter in all the treatments but remained generally higher than the control until the end of the second growing season. Nitrate-N concentrations in lysimeters were below water quality standard limits in all the treatments applied at the rate of 225 kg PAN ha^{-1}. Accumulation of N, C, and Ca in the forest floor was well correlated with the amount of biosolids applied on each treatment. The surface

application of different type of biosolids had minimal impact upon total N and C in the mineral soil. Increasing application rates of anaerobically digested biosolids directly increased soil N availability and mineralization. Nitrate-N concentrations in lysimeters were above water quality standards limits during several months in the AD900 and AD1800 treatments. Significant differences in the forest floor total N, C and Ca were observed with increasing application rates of biosolids. Total C accumulation was significantly higher in the forest floor in the AD1800 treatment. However, we observed no effect on soil total C with increasing application rates of biosolids. We found that biosolids application during spring significantly increased soil extractable N, N mineralization, NO_3-N leaching, and total C in the mineral soil in comparison to the fall application. Fall application significantly increased NH_4-N leaching and soil extractable Ca. We observed no significant effect on ion exchangeable N measured on membranes, total N, C, Ca, and pH measured in the forest floor, and soil total N and pH in the mineral soil. Our results demonstrated that permitted surface application of biosolids at the rate of 225 kg PAN ha^{-1} in a loblolly pine plantation increased surface soil N availability without increasing the potential for NO_3-N groundwater pollution.

Acknowledgements

I would like to express my gratitude to my committee members, Dr. Thomas Fox, Dr. Mike Aust, Dr. Lee Daniels, Dr. James Burger, and Dr. Greg Evanylo for all their contributions and unique perspectives to develop my Ph.D. program. Thank you to all of them for giving me strong views about applied science. I am especially grateful to Dr. Fox for giving me the opportunity to come to Virginia Tech. Tom has been an exceptional advisor and friend, and a great inspiration about dedication and passion for science and education.

I would like to extend my gratitude to David Mitchem, Colleen Carlson, Tal Roberts, and Daniel Evans for all their expertise and time. Kathie Hollandsworth, Sue Snow, and Tracey Sherman for all the administrative support. I also want to thank all the undergraduate helpers Ryan Blitch, Jessica Coker and Andrew Sucre. Thanks to all my friends in Virginia Tech for Eric Sucre, Bradley Miller, Pipa Elias, Aaron Pratt, Claudia Cotton, Kirill Kostyanovsky and Charley Kelly. I would like to especially thank Aaron Pratt for his helped with all the fieldwork involved in this project and for all the conversation.

Finally, I am especially grateful to my mother for their dedication and support through all this years in the U.S.A. I would like to thanks to my sisters Mariana and Marcela, and all my friends from Chile for all the long distance support.

The Metropolitan Washington Council of Governments, Becas Presidente de la Republica-Chilean government, the Forest Nutrition Cooperative and Pontificia Universidad Catolica de Chile provided funding for this project. Synagro Technologies, Inc. assisted in the installation and maintenance of the study.

Dedication

I dedicated this work to my mother Cira and my grandmother Yolanda, for all their love and support through all these years.

Table of Contents

Chapter 4: Nitrogen Leaching Following Application of Biosolids in a Loblolly Pine (*Pinus taeda* L.) Plantation in the Virginia Piedmont

Chapter 5: Short-term Effects of Biosolids Application on Nitrogen and Carbon pools in a Loblolly Pine (*Pinus taeda* L.) Plantation

List of Tables

List of Figures

Chapter 1
Introduction

1.1. Justification

Biosolids are solid or liquid materials produced during the treatment of sewage sludge that has been sufficiently processed to allow land application (Evanylo 1999). The term biosolids was introduced by the wastewater treatment industry in the early 1990s, and lately incorporated by the U.S. environmental protection agency (USEPA), to distinguish high quality, treated sewage sludge from raw sewage sludge or from sewage sludge containing large amounts of pollutants. Approximately 7.2 million dry Mg of biosolids were beneficially used or disposed in the USA in 2004. From this total, about 55% were land applied for agriculture, forestry, or land reclamation purposes (Goldstein 2007).

In Virginia, 224000 dry metric tons of biosolids were land applied during 2004, nearly all of it in agricultural land. From the total biosolids applied, about 52000 dry metric tons of biosolids were generated in Virginia wastewater treatments plants, and the remaining difference came from Maryland and Washington D.C. (Beech et al. 2007). Most of biosolids have been applied in agricultural land close to the different biosolids sources, however, decreasing availability of agricultural land suitable for biosolids application in eastern Virginia caused by urban expansion in the Washington-Richmond-Norfolk corridor, may limit ongoing land application programs.

Forestland in the Piedmont and Upper Coastal Plain of Virginia provides an alternative location for land application of biosolids. Published research shows that land application of treated municipal and industrial wastewater on forestland has been utilized successfully at various locations in the United States for over 30 years (Cole et al. 1986). This research has

concluded that land application of biosolids to forestland is one of the most cost-effective and environmentally sound processes for the recycling biosolids (Bastian 1986).

Although land application of biosolids is considered a beneficial reuse of wastewater treatments residuals as a source of nutrients for plant growth, there are environmental risks and public concerns associated with land application, like biosolids odor, heavy metals accumulation, groundwater pollution, and pathogens. As pointed out by Nutter and Red (1986), effective design criteria are required for successful forestland application systems. Issues such as loading rate, nutrient assimilation rates in the ecosystem, nutrient losses, and growth response for various types of biosolids must be addressed. It may not be possible to simply extrapolate agricultural land application practices to forest settings and incorporate the ability of the forest soil to assimilate nutrients and affect nutrient bioavailability over time. Additional research on the growth response and environmental impact of biosolids application in forest ecosystems in the Virginia Piedmont is warranted in order to increase the understanding of biosolids properties and forest site characteristics.

1.2. Major Objectives

The major objectives of this research are to:

1. Compare the impact of different types of biosolids on N availability, mineralization, leaching and organic matter accumulation over time.
2. Compare the impact of different application rates on N availability, mineralization, leaching and accumulation over time, and

3. Compare the impact of different season of application of biosolids on N availability, mineralization, leaching and accumulation on the forest floor over time.

1.3. Hypotheses Tested

We will achieve these objectives by testing the following null hypothesis:

1. Ho: Different types of biosolids, application rates, and season of application of biosolids do not change surface soil N availability.
2. Ho: Different types of biosolids, application rates, and season of application of biosolids do not change surface soil N mineralization.
3. Ho: Different types of biosolids, application rates, and season of application of biosolids do not affect N leaching.
4. Ho: Different types of biosolids, application rates, and season of application of biosolids do not change N and carbon accumulation on the forest floor and mineral soil after surface application of biosolids.

1.4. References

Bastian, R.K. 1986. Overview on sludge utilization. P. 7-25 in The forest alternative for treatment and utilization of municipal and industrial wastes, Cole, D.W., C.L. Henry, and W.L. Nutter (eds.). University of Washington Press, Seattle and London.

Beech, N., K. Crawford, N. Goldstein, G. Kester, M. Lono-Batura, and E. Dzieyk. 2007. A national biosolids regulation, quality, end use and disposal survey: Final report. North East Biosolids and Residuals Association (NEBRA). 30.

Cole, D.W., C.L. Henry, and W.L. Nutter. 1986. The forest alternative for treatment and utilization of municipal and industrial wastes. University of Washington Press, Seattle and London. 582 p.

Evanylo, G.K. 1999. Agricultural Land Application of Biosolids in Virginia: Managing Biosolids for Agricultural Use. Crop and Soil Environmental Sciences Publication 452-303.

Goldstein, N. 2007. Biosolids management trends in the U.S. BioCycle: Journal of Composting & Organic Recycling 48:9.

Nutter, W.L., and J.T. Red. 1986. Future directions: wastewater application P. 55-61 in The forest alternative for treatment and utilization of municipal and industrial wastes, Cole, D.W., C.L. Henry, and W.L. Nutter (eds.). University of Washington Press, Seattle and London.

Chapter 2
Literature Review

2.1. Introduction

Biosolids are solid or liquid materials produced during the treatment of sewage sludge that have been sufficiently processed to allow land application of these materials as dictated by the regulatory requirements for land application as defined in the Code of Federal Regulations (Part 503) (N.R.C. 2002). The term biosolids was developed in the 1990s to differentiate high quality, treated sewage sludge from raw sewage sludge or from sewage sludge containing large amounts of pollutants. Biosolids are removed from wastewater during the treatment process and have been modified chemically and physically to improve their handling characteristics, improve their suitability for use as a nutrient material, and reduce the environmental and public health concerns associated with land application. Of the 16583 estimated publicly owned treatment works (POTW) in the United States, approximately 68% land-apply biosolids. Approximately 7.2 million dry metric tons of biosolids were beneficially used or disposed in the U.S.A during 2004. From this total, about 55% were applied to land for agronomic purposes, and a lower proportion was applied to forestland and land reclamation areas (Beech et al. 2007; Goldstein 2007) .

2.2. Regulation of Land Application of Biosolids in Virginia

Land application of biosolids is regulated at the federal level by the US Environmental Protection Agency (USEPA 1994). The regulations governing the land application of biosolids were established by the USEPA in 1993 in the Code of Federal Regulations, Title 40 (Part 503) under Section 405 (d) of the Clean Water Act. The Part 503 rule established management practices for land application of biosolids; setting concentration limits and loading rates for

specific chemicals and the treatment and use requirements designed to control and reduce pathogens or disease vectors.

Biosolids are divided into two classes on the basis of pathogen content: Class A and Class B. Class A biosolids are treated to reduce the presence of pathogens below detectable levels. These materials can be used without any pathogen-related restrictions. They can be sold to the general public and applied at sites where there is public access. Class B biosolids are treated to reduce pathogens, but still contain detectable levels; therefore, Class B biosolids have site restrictions designed to minimize the potential for human and animal exposure until environmental factors such as heat, sunlight or desiccation have reduced the pathogens further. These materials cannot be sold to the general public or used at sites where there is public use.

In Virginia, the Department of Environmental Quality (DEQ) regulates biosolids land application since 2008. Previously, land application of biosolids was under the regulatory oversight of the Virginia Department of Health (VDH). In 1995, the VDH adopted the Biosolids Use Regulations 12 VAC 5-585, which incorporates into Virginia law the federal regulations established under the Part 503 rule. These regulations were amended in 1997. In several aspects, the Virginia regulations are stricter than those at the federal level (U.V.A. 1997) . The current key provisions of the Virginia regulations include: 1) site specific permits valid for 5 years must be obtained from DEQ; 2) required buffers along streams and water body are generally wider; 3) nutrient management plans are required for land application within the Chesapeake Bay and for sites receiving frequent applications; and 4) application rates must be based on the agronomic rate for and soil pH.

Approximately 380000 ha of Virginia's more than 3.2 millions of ha are permitted for the land application of biosolids (Leone 2005). The permitted sites include cropland, pasture and hay

land, and forestland. Among these different uses, spreading on pastureland appears to be the most common.

The major source of out-of-state biosolids is the DC-Water and Sewer Authority Blue Plains wastewater treatment plant (Washington, DC). Biosolids from the Blue Plains facility accounted for roughly 60 % of the out-of-State biosolids applied in Virginia in 2002. Other out-of-state sources have included treatment plants in Maryland, New Jersey, New York, and Pennsylvania (VDH 2004).

2.3. Beneficial Aspects of Land Application of Biosolids

Land application of biosolids falls under beneficial reuse provisions because they contain plant essential nutrients that may benefit plant growth when applied under the appropriate circumstances (Kroiss 2003). The growth, yield, quality and health of crop plants typically are better when optimal levels of plant essential nutrients are available in the soil. Land application of biosolids are mainly executed in agricultural sites where food crops and nonfood crops are grown (Beech et al. 2007). Nonagricultural sites include forests, rangeland, mine reclamation land, and public use sites such as parks and golf courses.

Biosolids function as slow-release fertilizers, releasing plant essential nutrients over time to the crop plants. Application of biosolids to agricultural land in Virginia is done principally to supply N, P, and, sometimes, lime (Evanylo 1999a). The biosolids application rates appropriate for use on agricultural land can be determined as follows; (1) Determine the nutrient requirements for the expected crop yield based on soil test values, (2) Calculate the biosolids application rates based on crop N and P requirements, or soil lime requirement as appropriate,

(3) Calculate supplemental fertilizer needs by subtracting the plant available N and P supplied by biosolids from the calculated nutrient requirements.

2.4. Nitrogen Forms and Availability

Nitrogen limits growth in most pine plantations, and N fertilization is needed to increase productivity of most forests (Fox et al. 2007). Although many soils contain large pools of N, less than 5% of the organic N in soils is mineralized annually, and the resultant available N may be used by soil microbes or by competing vegetation (Vitousek et al. 1983).

Understanding the behavior of N in the soil system helps to maximize forest productivity while reducing the potential negative impacts of N fertilization on the environment. Nitrogen can be found in soil in various forms, with 95% or more present as organic N (Tisdale et al. 1985). The inorganic forms of N, nitrate (NO_3-N) or ammonium (NH_4-N) are components of the complex nitrogen cycle and have many fates in the ecosystem. They can leach through the soil profile, volatilize, adsorb to cation or anion exchange site, be immobilized by microbes, or be taken up by plants (Tisdale et al. 1985).

The amount of a specific form of N present at a given time depends on physical, chemical, biological and environmental factors that affect N transformations and losses from the soil (Boyle 1990; Bramryd 1981; Gilmour et al. 2003). Some of these factors are soil water content, pH, temperature, soil aeration, and microbial activity, presence of plants, liming, and applied fertilizers. Changes in N availability following biosolids applications to forest can be attributed to biosolids properties, and how they affect tree N uptake, leaching, ammonia volatilization, and denitrification, immobilization and mineralization of the organic compounds

(Hallett et al. 1999; Magesan et al. 1998; Medalie et al. 1994; Robinson and Polglase 2000; Wang et al. 2004).

Nitrogen can be added to the soil through organic matter mineralization, also as inorganic or organic fertilizer (Bosatta and Berendse 1984; Maimone et al. 1991), or through rainwater containing NH_4-N and NO_3-N. It may also be fixed by lightning, combustion, industrial, and biological processes by which molecular nitrogen (N_2) is combined with H_2 or O_2 (Brumme and Khanna 2008).

Mineralizable N is defined as the active fraction of soil organic N that can be converted to NO_3-N and NH_4-N through microbial activity (Maimone et al. 1991). Nitrogen mineralization from biosolids varies greatly between different sources of material (Wang et al. 2004) , and once biosolids are applied, the N mineralization and/or immobilization is influences by soil properties and temperature (Barbarika et al. 1985). Microorganisms will immobilize NH_4-N or NO_3-N in the soil if there is a high C:N ratio in decomposing organic matter (Tisdale et al, 1985).

In several type of agricultural and forest systems, nitrification occurred after fertilization and was favored by the large supply of soil NH_4-N. Nitrification is also affected by the soil pH, the presence of oxygen, soil moisture contents and temperature. It occurs rapidly, and is very common, since the environmental factors that encourage plant growth also benefit the nitrifying organisms (Katterer et al. 1998; Vitousek and Matson 1984). Nitrogen can be lost from waterlogged or flooded soils by denitrification. This is a process that takes place under anaerobic conditions. The lack of oxygen may cause certain bacteria to shift from aerobic respiration where O_2 is used as the terminal electron acceptor during organic matter decomposition. In anaerobic conditions NO_3-N serves as a terminal electron accept or which converts the NO_3-N to N_2. Denitrification, a primary process in N reduction, can also occur in wet forest soils, such as those

in forested riparian buffers (Lowrance et al. 1985). Losses by volatilization of ammonia (NH_3) usually happen from surface application of NH_4-N fertilizers. This process is negligible in acid or neutral soils, but are critical when biosolids are surface applied to forest (Robinson and Polglase 2000).

2.5. Potential Environmental Impacts of Land Application of Biosolids

Although land application of biosolids is considered a beneficial reuse of nutrients, there are environmental risks and public concerns associated with biosolids (Basta 1995; O'Connor et al. 2005). The most noticeable public concern with land application of biosolids is odor (Evanylo 1999c). Even when properly treated and applied, the odors from biosolids can be objectionable. There are also public concerns with large amount of truck traffic required to transport biosolids to the application site. Health risks associated with exposure to biosolids are also a concern, particularly with Class B biosolids that contain detectable levels of pathogens (Pepper et al. 2008). The leaching of regulated trace elements is usually not a significant concern because biosolids typically contain concentrations that are well below established threshold levels; however, biosolids contain N that may leach from the site following land application at rates that exceed the nutrient requirements of the vegetation (Basta 1995; USEPA 2000). Surface runoff may also carry excess nutrients to surface waters. These nutrients may contribute to nonpoint pollution and could lead to eutrophication of receiving waters (Penn and Sims 2002). In Virginia, nutrient management plans are required for land application projects in the Chesapeake Bay Watershed (U.V.A. 1997). Buffer strips adjacent to streams and lakes ranging from 35 to 100 feet are required on land spreading sites to protect water quality (U.V.A. 1997). The potential

negative impacts of land application of biosolids can be minimized through proper management practices.

In 1974, Congress passed the Safe Drinking Water Act, which requires the USEPA to determine safe levels of chemicals in drinking water. These are based on potential health risks. The Maximum Contaminant Level (MCL) for N as nitrate allowed in drinking water is 10 mg L^{-1} (USEPA 1986). High NO_3-N levels contribute to the eutrophication of surface waters (Smith et al. 1999). This results in a high rate of fish death and a decrease in water quality (Boesch et al. 2001; Forsbeg 1998). Improper N fertilizer management has been identified as a major contributor to excess NO_3-N concentrations in ground water in the Chesapeake Bay area (Boesch et al. 2001).

Nitrogen becomes a concern to water quality when it is leached in the form of NO_3-N. When water moves through the soil profile it may carry NO_3-N with it, allowing it to reach ground water. Conditions that promote percolation also increase NO_3-N leaching, since NO_3-N it is very soluble in water and highly mobile.

High NO_3-N concentrations in the soil, along with intense precipitation or excess irrigation on a highly permeable soil will result in NO_3-N leaching losses. Most N in biosolids is in organic form. The organic N needs to be mineralized before it becomes available for plant uptake. The resulting NH_4-N may be oxidized to NO_3-N through nitrification (Jussy et al. 2004; Simmons et al. 1996).

To minimize the risk of contamination of water bodies with N, it is important to match the pattern of biosolids application and N mineralization to the assimilating capacity of the forest ecosystem (Magill et al. 2000; Strahm and Harrison 2007; Vitousek and Matson 1984). Forest soil properties can reduce the negative impact of a potential nutrient leaching or overland flow

after biosolids application (Wang et al. 2004). The forest floor in pine plantations enhances infiltration and percolation of rainfall and reduces overland flow and the potential for nutrient loss in runoff (Aust 1994; Lowrance et al. 1984).

High rates of application of biosolids may result in excessive leaching of NO_3-N and captions (Aschmann et al. 1992; Brockway and Urie 1983; Harrison et al. 1995). Forest ecosystems are highly buffered with respect to the amount of nutrients lost to stream or ground water after nutrient addition. With the exception of forests saturated with N due to atmospheric deposition and low soil buffering capacity, fertilized forests generally release minimal N or P to stream water. In the Pacific Northwest, large applications of N (700 kg N ha^{-1}) and P (500 kg P ha^{-1}) to Douglas-fir [*Pseudotsuga menziesii (Mirbel*)] stands in the form of municipal biosolids resulted in minimal impacts to stream water quality (Grey 2002). Biologically available P and N were the same before and after biosolids application. Although NO_3-N concentrations doubled after biosolids application, they remained less than 1 mg L^{-1}.

Levels of soil nutrient leaching decrease if biosolids are applied to areas with significant potential for plant uptake or soil immobilization (McLaren et al. 2007). Nutrient uptake by tree roots in the surface soil is rapid under most conditions, which coupled with high rates of evapotranspiration, decreases the potential for leaching (Wells et al. 1986). However, these potential problems could be less important in forests when there is a forest floor that favored nutrient immobilization (Bengtsson et al. 2003; Vitousek et al. 1992) and the retention of ions on the soil exchange complex (Pritchett and Fisher 1987; Strahm and Harrison 2006). Soils from the southeast Piedmont have chemical properties that could reduce the leaching potential because of the high nitrate sorption level (Eick et al. 1999; Strahm and Harrison 2007; Toner et al. 1989).

Nitrate-N sorption is influenced by soil pH, Al and Fe oxides levels in the soil (Strahm and Harrison 2007).

2.6. Application of Biosolids to Forests

Applications of biosolids to improve productivity have been widely use in agriculture. Land application of treated municipal and industrial wastewater on forestland has been utilized successfully at various locations and forest type around the world (Chapman-King et al. 1986; Henry et al. 1993). These researches have concluded that land application of biosolids to forestland one of the most cost-effective and environmentally sound processes for the recycling biosolids. Despite of the recognize benefits of biosolids application, less than 1% of the biosolids generated in the U.S. is applied to forest land (Goldstein 2007).

Henry et al. (1994) listed several benefits of biosolids application on forest land; (1) increase nutrient availability in limited forest sites, (2) since forest land are normally far from populated areas, there are less concerns and pressure about public health concerns and land application regulations, (3) forest soils have the potential to immobilize larger amount of nutrients and,(4) the extensive amount of forest land available for biosolids application. However, the authors also recognized some forest specific potential conflicts with recreational uses, and forest wildlife.

2.7 Types of Biosolids

There are several different types of biosolids used in land application that are generated from different stabilization process (Rowell et al. 2001; Smith et al. 1998; Sommers 1977). Lime stabilized material is the most commonly use in the Mid Atlantic region, and consists of solid

material that is stabilized through the addition of alkaline material. The rise in biosolids pH decreases biological activity, reducing the pathogens and odor levels (Little et al. 1991). It also immobilizes metals as long as pH levels stay high. Anaerobically digested material is biologically stabilized through conversion of organic matter to carbon dioxide, water, and methane. This reduces the biodegradable content and the quantity of material (Evanylo 1999b). Pelletized biosolids are a more highly processed material that requires greater initial production. The biosolids are heated at different temperatures and then dried to produce pellets (Matsuoka et al. 2006). The process used to produce pellets substantially reduces microbial populations, including pathogenic species, which changes the biosolids material from class B to class A in USEPA regulations (N.R.C. 2002).

Rates of biosolids are usually determined based on the N required by the crop (Evanylo 1999a). As in agricultural systems, biosolids supply plant essential nutrients that are deficient in most forest ecosystems, particularly N and P. The Forest Nutrition Cooperative has documented Loblolly pine growth increases of 30% with 224 kg of N and 28 kg of P fertilizer per ha (Fox et al. 2007).

Only a portion of the total amount of N contained in biosolids is present in a plant available form. Organic N compounds must be broken down and mineralized before the N is available to the plants. The amount of N mineralized over time varies depending on the properties of the biosolids and the land application site (Cogger et al. 2004; Eldridge et al. 2008; Gilmour 2003). Evanylo (1999a) describes a simple method that can be used to calculate plant available nitrogen (PAN) from which the agronomic biosolids application rate can be determined. In specific circumstances, other properties of the biosolids must be used to determine appropriate application rates. For example, when using lime-stabilized materials, the

impact of the biosolids on soil pH may determine the maximum application rate. Most agronomic crops require soils with pH values between 5.8 and 6.5. Pine forests typically require that soil pH values remain below 6.0; thus, the maximum application rate must be based on the lime equivalent of the biosolids. On most soils, the P contained is biosolids rapidly sorbed on Fe and Al oxyhydroxide surfaces. In these soils, P concentrations in soil solution remain relatively low, and P leaching losses are minimal. In sandy soils with low amounts of Fe and Al, P sorption capacity is low and leaching of P may be significant if large quantities of P are applied in biosolids. Thus, on these soils, P loading rates may determine the maximum amount of biosolids that can be land applied. Methods have been developed to determine the agronomic P rate and lime requirement for biosolids applications (Evanylo 1999a). In forest plantations, the extensive root system and accumulation of organic matter in biomass promotes retention of N and P in forest ecosystems (Jorgensen et al. 1980; Swanston et al. 2004). Managed pine forests tend to grow on nutrient deficient soils and, thus, are effective nutrient sinks.

It has been hypothesized that additions of municipal biosolids will promote long-term improvements in site quality and productivity that exceed those of chemical fertilization (Brockway et al. 1986; Henry et al. 1993). These improvements are a consequence of prolonged mineralization of N and other nutrients and improved moisture retention. Most of the biosolids decomposition studies have been conducted in agriculture sites or laboratory conditions. Rates of decomposition of soil organic matter (SOM) vary widely among biosolids (Gilmour et al. 2003), with much of this variation attributable to the influence of soil temperature, moisture, texture, and disturbance. Substantial uncertainties remain in estimates of soil organic matter turnover times affected by the nutrient status.

There have been few direct comparisons of tree growth responses to biosolids and conventional inorganic fertilization in forest. Prescott and Brown (1998) found no evidence of sustained growth response to biosolids in relation with conventional fertilizer, on the basis of 5-year growth responses. In later measurements Prescott and Blevins (2005) reported for the same study, that biosolids appear to be as effective as chemical fertilizers in promoting growth of western red cedar (*Thuja plicata*), western hemlock (*Tsuga heterophylla*), and amabilis fir [*Abies amabilis*].

The growth response following biosolids application to loblolly pine forests has been inconsistent. (McKee et al. 1986) showed that liquid, but not solid; biosolids applications increased the growth of loblolly pine plantations. In young plantations, the increased competition from weeds whose growth was stimulated by the sludge application detrimentally affected the growth of the pine trees (Brockway 1983).

2.8. References

Aschmann, S.G., M.S. McIntosh, J.S. Angle, and R.L. Hill. 1992. Nitrogen movement under a hardwood forest amendd with liquid waste-water sludge. Agriculture Ecosystems & Environment 38(4):249-263.

Aust, W.M. 1994. Best management practices for forested wetlands in the southern Appalachian region. Water, Air, and Soil Pollution 77:457-468.

Barbarika, A., L.J. Sikora, and D. Colacicco. 1985. Factors affecting mineralization of nitrogen in sewage-sludge applied to soils. Soil Science Society of America Journal 49(6):1403-1406.

Basta, N.T. 1995. Land application of biosolids : a review of research concerning benefits, environmental impacts, and regulations of applying treated sewage sludge. Oklahoma Agricultural Experiment Station : Center for Agriculture and the Environment, Division of Agricultural Sciences and Natural Resources, Oklahoma State University, [Stillwater]. xii, 59 p. p.

Beech, N., K. Crawford, N. Goldstein, G. Kester, M. Lono-Batura, and E. Dzieyk. 2007. A national biosolids regulation, quality, end use and disposal survey: Final report. North East Biosolids and Residuals Association (NEBRA). 30.

Bengtsson, G., P. Bengtson, and K.F. Mansson. 2003. Gross nitrogen mineralization-, immobilization-, and nitrification rates as a function of soil C/N ratio and microbial activity. Soil Biology & Biochemistry 35(1):143-154.

Boesch, D.F., R.B. Brinsfield, and R.E. Magnien. 2001. Chesapeake Bay eutrophication: Scientific understanding, ecosystem restoration, and challenges for agriculture. P. 303-320 in Annual Meetings of the American-Society-of-Agronomy, Anaheim, California.

Bosatta, E., and F. Berendse. 1984. Energy or nutrients regulation of decomposition-Implications for the mineralization immobilization response to pertubations. Soil Biology & Biochemistry 16(1):63-67.

Boyle, M. 1990. Biodegradation of land-applied sludge. Journal of Environmental Quality 19(4):640-644.

Bramryd, T. 1981. Comparative studies of nitrogen mineralization in forest soils fertilized with fluid and dewatered sewage sludge. P. 475-483.

Brockway, D.G. 1983. Forest Floor, Soil, and vegetation responses to sludge fertilization in Red and White-Pine Plantations. Soil Science Society of America Journal 47(4):776-784.

Brockway, D.G., and D.H. Urie. 1983. Determining Sludge Fertilization Rates for Forests from Nitrate-N in Leachate and Groundwater. Journal of Environmental Quality 12(4):487-492.

Brockway, D.G., D.H. Urie, P.V. Nguyen, and J.B. Hart. 1986. Wastewater and sludge nutrient utilization in forest ecosystems. P. 221-245. P. 221-245 in The forest alternative for wastewater and sludge nutreatment and utilization of municipal and industrial wastes, D.W., C., C. Henry, and N. W.L. (eds.). University of Washington Press, Seattle.WA.

Brumme, R., and P.K. Khanna. 2008. Ecological and site historical aspects of N dynamics and current N status in temperate forests. Global Change Biology 14(1):125-141.

Chapman-King, R., T.M. Hinckley, and C.C. Grier. 1986. Growth response of forest trees to wastewater and sludge application. P. 209-220.

Cogger, C.G., A.I. Bary, D.M. Sullivan, and E.A. Myhre. 2004. Biosolids processing effects on first- and second-year available nitrogen. Soil Science Society of America Journal 68(1):162-167.

Eick, M.J., W.D. Brady, and C.K. Lynch. 1999. Charge properties and nitrate adsorption of some acid southeastern soils. Journal of Environmental Quality 28(1):138-144.

Eldridge, S.M., K.Y. Chan, Z.H. Xu, C.R. Chen, and I. Barchia. 2008. Plant-available nitrogen supply from granulated biosolids: implications for land application guidelines. Australian Journal of Soil Research 46(5):423-436.

Evanylo, G.K. 1999a. Agricultural Land Application of Biosolids in Virginia: Managing Biosolids for Agricultural Use. Crop and Soil Environmental Sciences Publication. 452-303. Virginia Cooperative Extension Service. Blacksburg, VA.

Evanylo, G.K. 1999b. Agricultural Land Application of Biosolids in Virginia: Production and Characteristics of Biosolids. Crop and Soil Environmental Sciences Publication. 452-301 Virginia Cooperative Extension Service. Blacksburg, VA.

Evanylo, G.K. 1999c. Agricultural Land Application of Biosolids in Virginia: Risks and Concerns. Crop and Environmental Sicences Publications. 452-304. Virginia Cooperative Extension Service. Blacksburg, VA.

Forsbeg, C. 1998. Which policies can stop large scale eutrophication? Water Science Technology 37(3):193-200.

Fox, T.R., H.L. Allen, T.J. Albaugh, R. Rubilar, and C.A. Carlson. 2007. Tree nutrition and forest fertilization of pine plantations in the southern United States. Southern Journal of Applied Forestry 31(1):5-11.

Gilmour, J.T., C.G. Cogger, L.W. Jacobs, G.K. Evanylo, and D.M. Sullivan. 2003. Decomposition and plant-available nitrogen in biosolids: Laboratory studies, field studies, and computer simulation. Journal of Environmental Quality 32(4):1498-1507.

Gilmour, J.T., Craig G. Cogger, Lee W. Jacobs, Gregory K. Evanylo, and Dan M. Sullivan. 2003. Decomposition and Plant-Available Nitrogen in Biosolids: Laboratory Studies, Field Studies, and Computer Simulation. Journal of Environmental Quality 32:1498-1507.

Goldstein, N. 2007. Biosolids management trends in the U.S. BioCycle: Journal of Composting & Organic Recycling 48:9.

Grey, M., and Chuck Henry. 2002. Phosphorus and Nitrogen Runoff from a Forested Watershed Fertilized with Biosolids. Journal environmental quality 31:926-936.

Hallett, R.A., W.B. Bowden, and C.T. Smith. 1999. Nitrogen dynamics in forest soils after municipal sludge additions. Water Air and Soil Pollution 112(3-4):259-278.

Harrison, R.B., C.L. Henry, D.W. Cole, and D. Xue. 1995. Long-term changes in organic matter in soil receiving application of municipal biosolids. P. 139-153 in 8th North American Forest Soils Conference, McFee, W.W., J.M. Kelly, and J.M. Bigham (eds.), Gainsville, Fl.

Henry, C.L., D.W. Cole, and R.B. Harrison. 1994. Use of municipal sludge to restore and improve site productivity in forestry - The pack forest sludge research program. P. 137-149 in IEA/BE Workshop on Ameliorative Practices for Restoring and Maintaining Long-Term Productivity in Forests, Vaxjo, Sweden.

Henry, C.L., D.W. Cole, T.M. Hinckley, and R.B. Harrison. 1993. The use of municipal and pulp and paper sludges to increase production in forestry. Journal of Sustainable Forestry 1(3):41-55.

Jorgensen, J.R., C.G. Wells, and L.J. Metz. 1980. Nutrient Changes in Decomposing Loblolly-Pine Forest Floor. Soil Science Society of America Journal 44(6):1307-1314.

Jussy, J.H., M. Colin-Belgrand, E. Dambrine, J. Ranger, B. Zeller, and S. Bienaime. 2004. N deposition, N transformation and N leaching in acid forest soils. Biogeochemistry 69(2):241-262.

Katterer, T., M. Reichstein, O. Andren, and A. Lomander. 1998. Temperature dependence of organic matter decomposition: a critical review using literature data analyzed with different models. Biology and Fertility of Soils 27(3):258-262.

Kroiss, H. 2003. What is the potential for utilizing the resources in sludge? P. 1-10 in International Conference on Wastewater Sludge as a Resource (BIOSOLIDS 2003), Trondheim, Norway.

Leone, P. 2005. Review of Land Application of Biosolids in Virginia. P. 116, Commission, J.L.A.a.R. (ed.). Commonwealth of Virginia, Richmond, Virginia.

Little, D.A., R.B. Reneau, and D.C. Martens. 1991. Lime-stabilized and chemical-fixed sewage sludges as lime amendments. Bioresource Technology 37(1):93-102.

Lowrance, R., R. Leonard, and J. Sheridan. 1985. Managing riparian ecosystems to control nonpoint pollution. J. Soil and Water Conservation 40(1):88-91.

Lowrance, R.R., T.J.F. Jr., O.H. Jr, R. Leonard, and L. Asmussen. 1984. Riparian forests as nutrient filters in agricultural watersheds. BioScience 34:374-377.

Magesan, G.N., C.D.A. McLay, and V.V. Lal. 1998. Nitrate leaching from a free-draining volcanic soil irrigated with municipal sewage effluent in New Zealand. Agriculture Ecosystems & Environment 70(2-3):181-187.

Magill, A.H., J.D. Aber, G.M. Berntson, W.H. McDowell, K.J. Nadelhoffer, J.M. Melillo, and P. Steudler. 2000. Long-term nitrogen additions and nitrogen saturation in two temperate forests. Ecosystems 3(3):238-253.

Maimone, R.A., L.A. Morris, and T.R. Fox. 1991. Soil-Nitrogen mineralization potential in a fertilized loblolly-pine plantation. Soil Science Society of America Journal 55(2):522-527.

Matsuoka, K., N. Moritsuka, T. Masunaga, K. Matsui, and T. Wakatsuki. 2006. Effect of heating treatments on nitrogen mineralization from sewage sludge. Soil Science and Plant Nutrition 52(4):519-527.

McKee, W.H., Jr., K.W. McLeod, C.E. Davis, M.R. McKevlin, and H.A. Thomas. 1986. Growth response of loblolly pine to municipal and industrial sewage sludge applied at four ages on upper coastal plain sites. P. 272-281.

McLaren, R.G., L.M. Clucas, T.W. Speir, and A.P.v. Schaik. 2007. Distribution and movement of nutrients and metals in a Pinus radiata forest soil following applications of biosolids. Environmental Pollution 147(1):32-40.

Medalie, L., W.B. Bowden, and C.T. Smith. 1994. Nutrient leaching following land application of aerobically digested municipal sewage sludge in a northern hardwood forest. Journal of Environmental Quality 23(1):130-138.

N.R.C. 2002. Biosolids applied to land : advancing standards and practices. National Academies Press. . xviii, 345 p.

O'Connor, G.A., H.A. Elliott, N.T. Basta, R.K. Bastian, G.M. Pierzynski, R.C. Sims, and J.E. Smith. 2005. Sustainable land application: An overview. Conference on Sustainable Land Application:7-17.

Penn, C.J., and J.T. Sims. 2002. Phosphorus forms in biosolids-amended soils and losses in runoff: Effects of wastewater treatment process. Journal of Environmental Quality 31(4):1349-1361.

Pepper, I.L., H. Zerzghi, J.P. Brooks, and C.P. Gerba. 2008. Sustainability of land application of class B biosolids. Journal of Environmental Quality 37:58-67.

Prescott, C.E., and L.L. Blevins. 2005. Eleven-year growth response of young conifers to biosolids or nitrogen and phosphorus fertilizer on northern Vancouver Island. Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 35(1):211-214.

Prescott, C.E., and S.M. Brown. 1998. Five-year growth response of western red cedar, western hemlock, and amabilis fir to chemical and organic fertilizers. Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 28(9):1328-1334.

Pritchett, W.L., and R.F. Fisher. 1987. Properties and management of forest soils. John Wiley, New York, NY.494 pp.

Robinson, M.B., and P.J. Polglase. 2000. Volatilization of nitrogen from dewatered biosolids. Journal of Environmental Quality 29(4):1351-1355.

Rowell, D.M., C.E. Prescott, and C.M. Preston. 2001. Decomposition and nitrogen mineralization from biosolids and other organic materials: Relationship with initial chemistry. Journal of Environmental Quality 30(4):1401-1410.

Simmons, J.A., J.B. Yavitt, and T.J. Fahey. 1996. Watershed liming effects on the forest floor N cycle. Biogeochemistry 32(3):221-244.

Smith, S.R., V. Woods, and T.D. Evans. 1998. Nitrate dynamics in biosolids-treated soils. I. Influence of biosolids type and soil type. Bioresource Technology 66:139-149.

Smith, V.H., G.D. Tilman, and J.C. Nekola. 1999. Eutrophication: impacts of excess nutrient imouts on freshwater, marine, and terrestrial ecosystems. . Environmental Pollution 100:179-196.

Sommers, L.E. 1977. Chemical composition of sewage sludges and analysis of their potential use as fertilizers. Journal of Environmental Quality 6(2):225-232.

Strahm, B.D., and R.B. Harrison. 2006. Nitrate sorption in a variable-charge forest soil of the Pacific Northwest. Soil Science 171(4):313-321.

Strahm, B.D., and R.B. Harrison. 2007. Mineral and organic matter controls on the sorption of macronutrient anions in variable-charge soils. Soil Science Society of America Journal 71(6):1926-1933.

Swanston, C., P.S. Homann, B.A. Caldwell, D.D. Myrold, L. Ganio, and P. Sollins. 2004. Long-term effects of elevated nitrogen on forest soil organic matter stability. Biogeochemistry 70(2):227-250.

Tisdale, S.L., W.E. Nelson, and J.D. Beaton. 1985. Soil fertility and Fertilizer.4th edition. Mcmillan. New York, NY. 754 pp.

Toner, C.V.I., D.L. Sparks., and T.H. Carski. 1989. Anion Exchange Chemistry of Middle Atlantic Soils: Change Properties and Nitrate Retention Kinetics. Soil Science Society of America Journal 53:1061-1067.

U.V.A. 1997. Land application of biosolids in Virginia: A Study prepared for the Virginia Department of Health. UVA Institute for Environmental Negotiation. 47.

USEPA. 1986. Quality criteria for water EPA-440/5-86-001. U.S. Environmental protection agency.Washington, DC.

USEPA. 1994. Biosolids recycling: beneficial technology for a better environment. EPA-832/R-94-009. U.S. Environmental protection agency. Washington, DC. 25 pp.

USEPA. 2000. Biosolids Technology fact sheet. Land application of biosolids. EPA 832-F-00-052. Office of water. U.S. Environmental protection agency. Washington, DC.9 pp.

VDH. 2004. Report of the Virginia Department of Health on the U.S. Environmental Protection Agency's response to the National Research Council's report pertaining to the land application of biosolids to the Governor and the General Assembly of Virginia. Commonwealth of Virginia, Richmond, Va. 20 p. p.

Vitousek, P.M., S.W. Andariese, P.A. Matson, L. Morris, and R.L. Sanford. 1992. Effects of harvest intensity, site preparation, and herbicide use on soil nitrogen transformations in a young loblolly pine plantation. Forest Ecology and Management 49(3-4):277-292.

Vitousek, P.M., K.v. Cleve, N. Balakrishnan, and D. Mueller-Dombois. 1983. Soil development and nitrogen turnover in montane rainforest soils on Hawai'i. Biotropica 15(3):268-274.

Vitousek, P.M., and P.A. Matson. 1984. Mechanisms of nitrogen retention in forest ecosystems: A field experiment. Science 225:51-52.

Wang, H.L., G.N. Magesan, M.O. Kimberley, T.W. Payn, P.J. Wilks, and C.R. Fisher. 2004. Environmental and nutritional responses of a Pinus radiata plantation to biosolids application. Plant and Soil 267(1-2):255-262.

Wells, C.G., C.E. Murphy, C. Davis, D.M. Stone, and G.J. Hollod. 1986. Effect of sewage sludge from two sources on element flux in soil solution of loblolly pine plantations. P. 154-165 in The Forest Alternative for Treatment and Utilization of Municipal and Industrial Wastes, Cole, D.W., C. Henry, and W.L. Nutter (eds.). University of Washington Press, Seattle and London.

Chapter 3

Soil Nitrogen Availability and Mineralization Following Biosolids Application to a Loblolly Pine (*Pinus taeda* L.) Plantation.

Abstract

Nitrogen (N) limits growth in most of southern pine plantations growing in the Piedmont. Application of biosolids as a source of N is a common practices on farms in Virginia, USA. However, little is known about how biosolids type, timing and rate affect N availability in pine plantations over time. The objectives of the study were (1) to determine changes in N availability and mineralization through time after one application of different type of biosolids, (2) to evaluate the effect of increasing application rate on N availability, and (3) to determine the effect of season of application of biosolids. The study was established in Virginia at a 17-year-old loblolly pine plantation in Amelia County, VA. Anaerobically digested (AD225), lime stabilized (LS225) and pelletized (Pellet225) biosolids, and urea plus diammonium phosphate (U+DAP225) were applied at a rate of 225 kg ha^{-1} Plant Available Nitrogen (PAN) between March 5th and 10th, 2006. Anaerobically digested biosolids were also surface applied at the rates of 900 kg PAN ha^{-1} and 1800 kg PAN ha^{-1} (AD900 and AD1800). Anaerobically digested biosolids at the rate of 900 kg PAN ha^{-1} was previously applied in November 5th, 2005 (AD900F).

Nitrogen availability, as measured by ion exchange membranes (IEM) significantly increased following surface application of biosolids and conventional fertilizer ($p < 0.001$). The average total extractable N (IEM-Nt) for the 20-month period was significantly higher in the Pellet225 treatment in comparison to the AD225 ($p = 0.0039$) and LS225 ($p = 0.01$) treatments. The average N mineralization was significantly higher in the Pellet225 than in the AD225 ($p = 0.0253$). NH_4-N concentration in ion exchange membranes (IEM-NH_4) immediately increased in

the Pellet225, AD225, and U+DAP225 treatment. Soil NO_3-N contributions (IEM-NO_3) to the total available N were greater in the AD225, and LS225 treatments. The AD900 and the AD18000 significantly increased IEM-Nt and N mineralization in comparison to the AD225 ($p < 0.0001$). Differences in IEM-Nt, IEM-NH_4, and IEM-NO_3 were not significant between the AD900F and AD900. The average N mineralization was significantly greater in the AD900S treatment than in the AD900F ($p = 0.0427$). Nitrogen release from biosolids depends on mineralization and the initial N content in biosolids. Application of biosolids to a loblolly pine plantation is a good source of N when they are surface applied to loblolly pine forests.

3.1. Introduction

Biosolids are solid or liquid materials produced during the treatment of sewage sludge that has been sufficiently processed to allow land application (Evanylo 1999a), following the regulatory requirements defined by USA Federal Regulations (Part 503) (N.R.C. 2002) . Approximately 7.2 million Mg of biosolids were produced in the year 2004. From this total, about 55% were applied to land as fertilizer for agronomic purposes, and in a lower proportion to forestland and land reclamation (Beech et al. 2007). In Virginia land application of biosolids to agriculture and forest land is becoming increasingly popular with about 136000 Mg land applied every year (VDH 2004).

Nitrogen (N) and phosphorus (P) limit growth in most southern pine plantations, and fertilization with urea and diammonium phosphate (DAP) is a regular forest management prescription (Albaugh et al. 2007). In loblolly pine (*pinus taeda*), volume growth increases of 30 % typically occur following fertilizer applications of 225 kg of N and 28 kg of P ha^{-1} (Fox et al. 2007). Even though the surface application of urea rapidly increases N availability, the effect is generally short lived.

Application of biosolids to forest increases essential nutrients, particularly N and P (Brockway et al. 1986; Henry et al. 1994; Jokela et al. 1990; Prescott and Blevins 2005). Typical dried biosolids contain approximately 4% N, with 0.65% NH_4-N and low or no NO_3-N (Evanylo 1999b). Several studies reported positive changes in soil nutrient availability and mineralization following biosolids application to forests (Brockway 1983; Hallett et al. 1999; Magesan et al. 1998; Medalie et al. 1994; Wang et al. 2004).

The N added as NH_4-N in the biosolids initially increases soil N availability, although a portion may be lost through volatilization (Henry et al. 1999; Robinson and Polglase 2000). Long term N availability is regulated by the mineralization of organic N in biosolids (Robinson et al. 2002). Different sources of biosolids mineralize N at different rates depending on their chemical and physical composition. Biosolids that go through anaerobic digestion, lime stabilization, heat treatment or composting will decompose at different rates (Parnaudeau et al. 2006; Rowell et al. 2001). For example, anaerobically digested biosolids had lower N mineralization rates than aerobically digested biosolids as a consequence of the stabilization processes (Garau et al. 1986). Certain biosolids treatments used to reduce pathogens, like lime addition, affect soil pH, reducing soil microbial activity and mineralization of organic N (Simmons et al. 1996).

Permitted rates of land application of biosolids are usually based on the N additions and therefore, need to consider the initial organic and inorganic N concentration, application methods, type of biosolids, and plant demand (Evanylo 1999a; Henry et al. 1999; USEPA 1995). High application rates directly increase N availability, and also the potential for NO_3-N leaching if the available N exceeds the plant requirements (Aschmann et al. 1992; Brockway and Urie 1983; Kelty et al. 2004). The risk of N leaching decreases if biosolids are surface applied due to

low nutrient incorporation (Gove et al. 2002; Jaynes et al. 2003), and when biosolids are applied to areas with a large nutrient demand like forest (Henry et al. 1994; McLaren et al. 2007).

There is little field data available on the changes in N availability and mineralization after surface application of different type of biosolids to pine plantations, and how this varies with application rate and season of application. The purpose of this study was to determine changes in available N and mineralization after surface application of several sources of biosolids to a 17-years-old loblolly pine plantation. We also evaluated the impacts of application rate and season of application on nitrogen availability. The specific objectives of the study were (1) to determine changes in N availability in the forest floor and surface mineral soil through time after one application of different type of biosolids, (2) to evaluate the effect of increasing application rates on N availability, and (3) to determine the effect of season of application on N availability.

3.2. Materials and Methods

3.2.1. Study Area

The study was established in the summer of 2005 in Amelia County northeast of Blackstone, VA (37° 13' N, 77° 48' W). The site is located in the Piedmont physiographic province and supported a 17-year-old loblolly pine plantation. The stand was thinned in 2005 using a combination of fifth-row removal and low thinning between the removed rows. The mean annual temperature is 14 °C, with a mean of 4 °C in January and 25 °C in July. The mean annual precipitation is 113 cm with July and August being the wettest. The mean snowfall is 30 cm concentrated between December and March (15-year period). The local climatologic recent records were obtained from the closest weather station in Blackstone, VA, which was approximately 40 km from the study site. During the month of the spring application (March

2006), precipitation was only 0.68 cm, which was below the historical average 8.1 cm for the area (Fig 3.1). Conditions were near normal throughout most of the spring and summer of the same year, while rainfall from August to November 2006 was greater than normal.

The soil at the site is the Appling series (Fine, kaolinitic, thermic Typic Kanhapludults). Slopes range from 2% to 5%. The thin Ap layer shows evidence of surface soil erosion caused by past agriculture practice, and in some areas is mixed with the argillic Bt horizon. Soil samples collected from 0 to 20 cm prior to treatments were analyzed for total C and N by combustion using a CNS analyzer (Elementar America Inc., Laurel, NJ), and Mehlich-1 extractable P, K, Ca, Mg using a ICAP-AES following the Virginia Cooperative Extension Methodology (Donohue and Heckendorn 1994; Mehlich 1953). Soil pH was determined in a 1:1 soil:water ratio. Soil cores were collected with a bulk density hammer and subsequently oven dried and weigh to measure bulk density (Table 3.1).

3.2.2. Experimental Design and Treatments

The experimental design was a randomized complete block design with four blocks and eight treatments comparing biosolids types, granular fertilizer, application rates, and season of application. Thirty-six plots of 0.45 ha (150 x 30 m) separated by a 30 m of untreated buffer area were established in September 2005. Three different types of biosolids were evaluated; lime stabilized, anaerobically digested, and pelletized. The anaerobically digested material was obtained from the AlexandriaVA, and Back River MD, wastewater treatment facilities, for the fall and spring application, respectively. The lime stabilized biosolids were obtained from the Blue Plains wastewater treatment facility DC. The pelletized biosolids were obtained from the Synagro pelletized facility in Back River MD. The granular fertilizer treatments were a mix of conventional urea-N (46-0-0) and DAP (18-46-0) obtained from Southern States Cooperative,

Inc., (Christiansburg, VA). Physical and chemical properties of the biosolids are presented in Table 3.2.

The biosolids were applied during two seasons, fall and spring. Anaerobically digested, lime stabilized and pelletized biosolids and a conventional inorganic fertilizer were applied at a rate of 225 kg PAN ha^{-1} (plant available N). In order to understand the consequences of increasing the rates on N availability, anaerobically digested biosolids were added at 900 and 1800 kg PAN ha^{-1}. These treatments were applied between March 5th and 10th, 2006. To test the impact of application timing (fall vs. spring), the anaerobically digested biosolids were added at 900 kg PAN ha^{-1} on November 5th, 2005. Treatments descriptions and final rates are listed in Table 3.3.

Biosolids application rates were estimated based on the average N concentration of samples analyzed during the six previous months before the beginning of the study, and corroborated with field samples. The treatments rates were based on PAN approach, determined according to recommendations established for VA when biosolids are surface applied (Evanylo 1999a).

$$\mathbf{PAN = NO_3\text{-}N + \mathit{Kvol}\ (NH_4\text{-}N) + \mathit{Kmin}\ (Org\text{-}N)}$$

Where:

PAN = Kg of Plant Available N dry^{-1} Mg biosolids

NO_3-N = Kg nitrate-N dry^{-1} Mg biosolids

Kvol = volatilization factor, or plant-available fraction of NH_4-N (lime stabilized = *0.25*, anaerobically digested = *0.5*)

NH_4-N =Kg ammonium-N dry^{-1} Mg biosolids

Kmin = mineralization factor, or plant-available fraction of Org-N (lime stabilized = *0.3*, and anaerobically digested = *0.2*)

Org-N = Kg organic-N dry^{-1} Mg biosolids (estimated by organic N = total Kjeldahl-N - NH_4-N)

Conventional granular fertilizer treatments were broadcast at the same time using a backpack spreader so the fertilizer evenly covered the forest floor. The biosolids were transported from each wastewater treatment plant, piled at the site and applied during the same day. Biosolids were surface applied using a skidder with a side discharge spreader that went through the previously thinned corridors. The biosolids were not tilled into the soil. Four plastic collection trays (60 x 20 cm) were installed in each plot prior to application. The biosolids collected in each treatment were weighed, and moisture content was determined after the application. Samples were collected from the trays in the field and transported to A&L Eastern Agricultural Laboratories (Richmond, VA) for routine biosolids test analysis for total and volatile solids (SM2540G), nitrate-N (SM4500- NO_3-F) (APHA 1998), pH according to SW 846-9045C (USEPA 2002), and calcium carbonate equivalent (CCE) using AOAC 955.01 (Kane 2000). Total Kjeldahl nitrogen (USEPA 351.3) and ammonium-N (USEPA 350.2) (USEPA 1983). Phosphorus (P), potassium (K), sulfur (S), calcium (Ca), magnesium (Mg), sodium (Na), iron (Fe), manganese (Mn), cadmium (Cd), copper (Cu), lead (Pb), molybdenum (Mo), nickel (Ni), and zinc (Zn) were measured according to SW846-6010B (USEPA 2002) (Table 2).

The competing herbaceous understory vegetation was removed chemically using a foliar application of 5% Round-up Pro ™ (Monsanto Co, St Louis MO) applied with a backpack sprayer in all plots during the summer of 2005 and 2006.

3.2.3. Nitrogen Availability

In situ monthly N availability was measured using ion exchange membranes (IEM) (Cooperband and Logan 1994; Subler et al. 1995). Cation and anion exchange membrane sheets (Ionics Inc., Watertown, MA) were cut into 10 by 10 cm pieces. Cation and anion membrane squares were kept separate, washed with de-ionized water, and soaked inside plastic carboys containing 1 M NaCl. In each plot, two pairs of cation and anion exchange membranes were randomly installed in a 45° angle through the surface soil to a depth of 10 cm, and horizontally between the forest floor layer and the topsoil. Individual membranes were carefully removed after four weeks, and a new set was randomly installed in each plot. Soil adhering to the membranes was removed, and membranes were stored on dry ice and transported to the lab were they were keep at 4 °C until extraction.

For extraction, membranes were placed in centrifuge tubes and 25 ml of 1 M KCl solution was added. Tubes were shaken on a reciprocal shaker for 1 hour and the solution was filtered through Whatman #42 into 25 ml scintillation vials. Extracts were frozen until analyzed colorometrically for NO_3-N (USEPA Method 353.2) and NH_4-N (USEPA Method 350.1) using a TRAACS 2000 Auto Analyzer (SEAL Analytical, Mequon, WI). After extraction, the membranes were cleaned and recharged with 1M NaCl solution, and stored until the next sampling period. Five sets of membranes were used through the period of the study, and they all were used the same number of times.

Ion exchange membranes provide an index of NO_3-N and NH_4-N availability, in both the forest floor and the surface mineral horizon following application of biosolids and conventional fertilizer. Trends in N concentrations from the exchange membranes were similar in the mineral soil and forest floor throughout the study; therefore N availability is reported as the sum of the N

extracted from the membranes located in the forest floor and the surface mineral soil. The concentrations of NH_4-N and NO_3-N extracted from the membranes are reported separately. In addition, the resultant sum of IEM-NO_3 and IEM- NH_4 is presented as total IEM-N_t. The total amount of N extracted from each type of membrane was divided by the amount of days that they were buried in the field, and expanded to 1 m^2 surface of membranes.

3.2.4. Field Nitrogen Mineralization

In situ N mineralization (N-min) was measured in each treatment plot using sequential coring methods (Gurlevik et al. 2004; Raison et al. 1987). Cores were made of polyvinyl chloride (PCV) pipe (3.8 diameter by 20.3 cm in length). Two cores were installed next two each other at a randomly located point in each measurement plot. Cores were inserted vertically into the mineral soil, to a depth of 15 cm. Caps were placed on the cores to exclude rainfall, and small lateral wholes were drilled to allow gas exchange within core. One core was immediately removed and placed in a cooler and returned to the lab for processing and analysis. The second incubation core remained in place for 4 weeks. After the incubation period, the core left in the field was also removed and processed in the same manner. In situ incubations were carried out every month. The soil was removed from the core and sieved with a #10 mesh screen. The sieved soil was stored at 4°C on plastic bags until extraction. At the time of soil extractions, 5 g. of field moist soil was dried at 105°C for 24 hours, and reweighed to determine moisture corrections. Another 5 g. sample of soil from the same sample was placed in a centrifuge tube with 50 ml of 2 M KCl and shaken on a reciprocating shaker for 1 hour. Samples were centrifuged for 10 minutes, filtered through Whatman #42 paper, and the extracts were transferred to scintillation vials. All extracts were analyzed colorometrically for ammonium (USEPA Method 350.1) and nitrate (USEPA Method 353.2) and using a TRAACS 2000 Auto Analyzer (SEAL Analytical, Mequon, WI). Nitrogen concentration measured from the mineralization cores was extrapolated

to kg per ha^{-1} based on surface soil bulk density and cores depth.

Soil net mineralization was determined based on the difference in NH_4-N + NO_3-N extracted from the initial soil core and the soil core left in the field during the period of exposure. Cumulative N mineralization was determined by summing the net mineralization from each incubation period.

Organic N mineralization in biosolids was calculated as the difference in N mineraliztion in the control plot and the biosolids amended plots. This assumes that soil N mineralization was not increased by addition of biosolids (Wang et al. 2003).

3.2.5. Statistical Analysis

All statistical analyses were conducted using SAS 9.1 statistical software (SAS Institute, Cary, NC). Analysis of variance (ANOVA) using the mixed models procedure with repeated measures were performed to test the effect of treatments, time, and the interactions on monthly IEM-N_t, IEM-NH_4, IEM-NO_3,, and N-min. Variance-covariance structures were examined to determine the best model for the repeated measures (Littell et al. 2006). Tests for normality, linearity, and constant variance of the residuals were performed. Logarithmic transformations were necessary to ensure the validity of the assumptions. Results are presented in untransformed values. When the treatment time interaction was significant ($P<0.05$ F test), Fisher-LSD test was conducted for multiple comparison at each sampling time at $\alpha = 0.05$. Selected contrasts were performed to compare the main treatment effect.

3.3. Results

3.3.1. Effect of Type of Biosolids

Different type of biosolids significantly affected IEM-NH_4, IEM-NO_3, IEM-Nt, and N-min. The ANOVA indicted that there was a significant treatment by time interaction for all the response variables analyzed (Table 3.4).

3.3.1.1. IEM-NH_4

The contrast analysis for the main effect indicated that the average IEM-NH4 in the Pellet225 treatment was significantly higher than the U+DAP225, LS225, and AD225 (Table 3.5). The U+DAP225 was significantly higher than the LS225 and the AD225, and no significant differences were detected between the LS225 and the AD225 (Table 3.5).

Biosolids application immediately increased IEM-NH_4 in the Pellet225, AD225, and U+DAP225 treatments relative to control and the LS225 (Fig 3.2.a, Table 3.6). The significant response persisted until December 2006 and then in June and July 2007. The peak response in IEM-NH_4 in the Pellet225 treatment was 23.11 mg m^{-2} day^{-1} in July 2006, 15.86 mg m^{-2} day^{-1} in May 2006 in the U+DAP225 and 9.56 mg m^{-2} day^{-1} in May 2006 in the AD225 treatment. From March 2006 to June 2006, there was no difference in IEM-NH_4 among the Pellet225, AD225, and the U+DAP225 treatments. However, IEM-NH_4 concentration in the AD225 and U+DAP225 treatments declined after July 2006 (Fig 3.2.a). During the period from July 2006 to December 2006, IEM-NH_4 was greater in the Pellet225 treatment than in the AD225 and the U+DAP225 treatment. After January 2007, IEM-NH_4 in the Pellet225, AD225 and U+DAP225 treatments were relatively low and similar to the control except in February, June, July, and August 2007 (Table 3.6).

The pattern of IEM-NH_4 in the LS225 treatment was different than the other biosolids treatments. Initially, IEM-NH_4 in the LS225 treatment was lower than the other biosolids treatments and close to the control treatment (Fig 3.2.a, Table 3.6). However, it increased in June 2006 and was significantly greater than the control through September 2006. IEM-NH_4 in the LS225 treatment peaked at 6.54 mg m^{-2} day^{-1} in July 2006. After that, it declined and by October 2006 was no longer significantly different than the control. As with the other biosolids treatments, IEM-NH_4 in the LS225 treatment increased sporadically during 2007 (Table 3.6).

3.3.1.2. IEM-NO_3

Average IEM-NO_3 availability in the plots treated with biosolids and conventional fertilizer were significantly higher than the control throughout the study (Table 3.5). The LS225, and the AD225 had the higher IEM-NO_3 concentration in comparison with the Pellet225, and the U+DAP225 treatment, but no significant differences were detected between the AD225 and the LS225 treatment (Table 3.5).

The application of biosolids significantly affected IEM-NO_3, however the impact occurred more slowly than it did for IEM-NH_4 (Fig 3.2.b, Table 3.7). Peak IEM-NO_3 concentration in the AD225 treatment were 8.69 mg m^{-2} day^{-1} in August 2006, 14.7 mg m^{-2} day^{-1} in the LS225 treatment in December 2006, 2.18 mg m^{-2} day^{-1} in the Pellet225 treatment in November 2006, and 3.42 mg m^{-2} day^{-1} in May 2006 in the U+DAP225 treatment.

IEM-NO_3 concentrations in the AD225 and the LS225 treatment followed a similar pattern (Fig 3.2.b). IEM-NO_3 concentration in both the AD225, and the LS225 were greater than in the control from March 2006 (LS225) to October 2006 (AD225) or November 2006 (LS225). In the AD225 treatment, IEM-NO_3 increased gradually and reached a peak in August 2006, and then remained elevated until October 2006, a period when IEM-NH_4 in the AD225 treatment was declining (Fig 3.2.a). IEM-NO_3 in the LS225 treatment also increased through time, but IEM-NO_3 concentration declined by September 2007.

IEM-NO_3 in the U+DAP225 and in the Pellet225 treatments followed a similar pattern and they were periodically greater than the control until December 2006, although the concentrations were relatively low (Fig 3.2.b, Table 3.7), and below the AD225 and the LS225 treatments (Table 3.7).

3.3.1.3. IEM-Nt

Treatments with both biosolids and U+DAP225 significantly increased IEM-Nt relative to the control treatment (Table 3.5). The mean average IEM-Nt response for the whole sampling period from the Pellet225 treatment was significantly higher than the LS225 and the U+DAP225. The LS225 and AD225 treatments were also significantly higher than the U+DAP225. The average IEM-Nt was significantly greater in the LS225 than in the AD225 treatment (Table 3.5).

IEM-Nt significantly increased after application of biosolids in the Pellet225, AD225, and U+DAP225 treatments relative to the control (Table 3.8). The cumulative peak in IEM-Nt concentration in the Pellet225 treatment was 24.13 mg m^{-2} day^{-1} in July 2006, the AD225 peak was 12.33 mg m^{-2} day^{-1} in May 2006, the LS225 had a peak of 16.67 mg m^{-2} day^{-1} August 2006, and the U+DAP225 treatment with a peak of 19.27 mg m^{-2} day^{-1} in May 2006.

Differences in total IEM-Nt throughout the different sampling points were explained by variable contribution of the IEM-NH_4, and the IEM-NO_3. The IEM-Nt concentrations in the Pellet225, AD225, and the U+DAP225 treatments immediately following biosolids application were driven by the IEM-NH_4, with peaks in the summer after application. They remained higher than the control until October 2006, in the AD225 and the U+DAP225 treatments (Fig 3.3.c). The IEM-Nt declined in the U+DAP225 treatment after July 2006, but remained significantly elevated in the AD225 and the Pellet225 until October 2006 and April 2007, respectively (Table 3.8). The contribution of the IEM-NO_3 to the total IEM-Nt was relevant in the AD225, and the LS225 treatments (Fig 3.2, Table 3.8). The LS225 IEM-Nt pattern was similar to the IEM-NO_3 with a later peak in August 2006, to then slowly drop to control levels by December 2006. After December 2006, all the treatments tended to decline but stayed greater than the control until July 2007 (Fig 3.2.c, Table 3.8).

3.3.1.4. N mineralization

The average response in soil N-min over the entire sampling period was greater in all the biosolids treatments and U+DAP225 than in the control. The Pellet225 was significantly larger than the AD225 and the U+DAP225 (Table 3.5). N mineralization (N-min) followed a similar pattern with the IEM-N results, with a greater early response in the Pellet225 and the U+DAP225 treatments, and a slower increased N-min in the LS225, and the AD225 treatments (Fig. 3.3, Table 3.9). The peak N-min was 23.3 kg ha^{-1} in the LS225 in August 2006, 38.3 kg ha^{-1} in the Pellet225 in August 2006, and 68.9 kg ha^{-1} in the U+DAP225 in July 2006, 37.1 kg ha^{-1} in the AD225 late in June 2007.

N-min in the U+DAP225 and the Pellet225 treatments were significantly higher relative to the control from April 2006 to September (U+DAP225) or October 2006 (Pellet225). After that, both treatments remained close to control levels (Table 3.9). The LS225 and the AD225 were significantly larger than the control from August to December 2006 (LS225) or September to October 2006 (AD225) (Table 3.9). The LS225 and AD225 had lower N-min relative to the Pellet225 and U+DAP225 (Fig. 3.3, Table 3.9). All the treatments had a second N-min peak during May and June 2007.

The total cumulative N-min during the 20-month measurements in the Pellet225 was 331.4 kg ha^{-1}, 298.4 kg ha^{-1} in the U+DAP225, 273.1 kg ha^{-1} in the LS225, and 230.2 kg ha^{-1} in the AD225 (Fig. 3.3). Organic-N mineralization from the biosolids in the first year following treatments application was 4.3%, 8.7%, and 45.5% of the total N added in the AD225, LS225, and the Pellet225, respectively. For the 20-month period N mineralization in biosolids was 10.1% in the AD225, 14.6% in the LS225, and 64.7% in the Pellet225 treatments.

3.3.2. Effect of Biosolids Application Rates

Increasing biosolids rates significantly increased the IEM-NH_4, IEM-NO_3, IEM-Nt, and N-min. There was a significant treatment by time interaction for the all the response variables (Table 3.4).

3.3.2.1. IEM-NH_4

IEM-NH_4 concentrations significantly increase with higher application rates of biosolids throughout the whole study. Contrast indicated that the AD1800 treatment had significantly higher concentrations of IEM-NH_4 in comparison with the AD900 and the AD225 treatments. The IEM-NH_4 concentration in the AD900S treatment was significantly higher than the AD225 treatment (Table 3.5).

The application of higher rates of biosolids significantly increased IEM-NH_4 during most of the sampling dates (Table 3.10). The maximum IEM-NH_4 concentration was 43.68 mg m^{-2} day^{-1} in March 2006 for the AD1800, 25.20 mg m^{-2} day^{-1} in April 2006 in the AD900, and 9.56 m^{-2} day^{-1} in May 2006 for the AD225 (Fig 3.4.a ,Table 3.10). The IEM-NH_4 declined through time at all three rates. IEM-NH_4 concentration in the AD900, and the AD1800 remained higher than the AD225 until March 2007 (AD900) or May 2007 (AD1800). In contrast, the IEM-NH_4 in the AD1800 and the AD900 remained at similar levels through most of the study (Fig 3.4.a, Table 3.10)

3.3.2.2. IEM-NO_3

Increasing application rates of biosolids directly increased the average IEM-NO_3 concentration in the three biosolids treatments throughout the whole study (Table 3.5).

Like the IEM-NH_4 response, the IEM-NO_3 concentrations also increased as biosolids application rate increased. However, IEM-NO_3 concentration increased more gradually than IEM-NH_4 concentrations (Fig 3.4.b). IEM-NO_3 concentrations peaked at 8.69 mg m^{-2} $day^{-1}$and

16.99 mg m^{-2} day^{-1} in August 2006 for the AD225, and AD900 treatment respectively. IEM-NO_3 concentration peaked one month later, in September 2006, in the AD1800 treatment at 32.84 mg m^{-2} day^{-1}. These peaks in IEM-NO_3 were later than the IEM-NH_4 peaks (Fig 3.4). IEM-NO_3 concentrations declined after July 2007, and there were no significant differences among the three application rates until November 2006. IEM-NO_3 concentrations in both the AD1800 and the AD900 treatment remained elevated throughout the study period relative to the AD225 treatment from November 2006 to April 2007 (Fig 3.4.b, Table 3.11).

3.3.2.3. IEM-Nt

The IEM-Nt concentration was most influenced by the concentration of IEM-NH_4 in all the treatments, with higher concentrations in the AD1800, relatively to the AD900 and the AD225 (Table 3.5). IEM-Nt from the AD1800 treatment peak was 47.71 mg m^{-2} day^{-1} in March 2006, and then slowly declined by the end of the second year (Fig 3.4.c). The peak in IEM-Nt in the AD225 was 12.33 mg m^{-2} day^{-1} in May 2006, and during the first year was significantly lower than the than AD1800 in March, May, and June 2006 (Table 3.12). The IEM-Nt were similar in the all the tree treatments from July to October 2006. The AD225 tended to decline after that and remained significantly lower than the AD1800 and the AD900 until April 2007. The IEM-Nt in the AD1800 was significantly higher than the AD900 treatment during March 2006, and then later in September 2007 (Table 3.12). The AD900 treatment had a peak of 28.38 mg m^{-2} day^{-1} during April 2006, and remained close to the AD1800 throughout most of the study (Table 3.12).

3.3.2.4. N-mineralization

Increasing application rates of biosolids increased the average N-min (Table 3.5). The mean estimated N-min during the whole study was significantly greater in the AD1800 and the AD900 in comparison to the AD225. However, there was no significant difference in the N-min between the AD1800 and the AD900 (Fig 3.5, Table 3.5).

The N-min for the AD1800, and the AD900 treatments were significantly greater than the AD225 immediately after treatments application in March 2006 (Table 3.13). The AD1800 and the AD900 N-min remained higher than the AD225 until October 2006 (AD1800) or February 2007 (AD900). The AD900 treatment was significantly greater than the AD1800 from November 2006 to January 2007. However, the AD1800 increased again in February 2007, and stayed elevated until May 2007 in comparison to the other two rates. The peak N-min was 173.2 kg ha^{-1} in the AD1800 during September 2006, 117.3 kg ha^{-1} in the AD900 in September 2006, and 37.1 kg N ha^{-1} in the AD225 late in June 2007 (Table 3.13). The total cumulative N-min in the AD1800 was 1196 kg ha^{-1}, 891 kg ha^{-1} in the AD900, and 230.2 kg ha^{-1} in the AD225.

Organic N mineralization in biosolids for first 12 months following treatments application was 5.4%, 26.1%, and 17.2% in the AD225, AD900, and the AD1800 treatment, respectively. For the 20-month period N mineralization was 10.3% in the AD225, 30.7% in the AD900, and 20.8% in the AD1800 treatments.

3.3.3. Effect of Season of Biosolids Application

3.3.3.1. IEM-NH_4, IEM-NO_3, and IEM-Nt

Season of application did not significantly increased total IEM-NH_4, IEM-NO_3, and total IEM-Nt after surface application of the same rate of anaerobically digested biosolids during November 2005 (fall), and March 2006 (spring) (Fig 3.6, Table 3.5, 3.14, 3.15, and 3.16).

3.3.3.2. N mineralization

Season of application of biosolids significantly affect N-min after surface application of the same rate of anaerobically digested biosolids. Nitrogen mineralization in the AD900S was greater than the AD900F (Table 3.5). N-min in both treatments tended to be similar throughout most of the sampling dates, with the exception of five months (Table 3.17).

Both applications followed the same mineralization pattern, with low levels after biosolids application to then slowly started to increased four (AD900F), and three months after application (AD900S). The peak N-min was 129.9 kg ha^{-1} in the AD900F seven months after application, and 161.5 kg ha^{-1} in the AD900S treatment, five months after application. Both treatments declined by December 2006, and were sporadically greater than the control during 2007 (Table 3.17). The cumulative N mineralization was 689 kg ha^{-1} in the AD900F and 892 kg ha^{-1} in the AD900S (Figure 3.7).

Organic N mineralization in biosolids for the first year following treatments application was 21.7%, and 26.1% in the AD900F, and in the AD900S, respectively. For the 20-month period N mineralization was 21.6% in the AD900F, and 30.8% in the AD900S treatment.

3.4. Discussion

3.4.1. Effect of Type of Biosolids on N Availability

Our results have demonstrated that N availability increases following land application of biosolids to forests, as shown in both the IEM-N availability and N mineralization data. Similar results were observed in other studies of biosolids application on *Pinus resinosa* Ait and *Pinus strobus* (Brockway 1983), *Pinus ponderosa* (Powers 1980), *Thuja plicata* (Prescott and Zabek 1997), mixed northern hardwoods forest (Hallett et al. 1999); and *Pinus radiata* (Wang et al.

2004). Increases in soil N availability have also been observed in loblolly pine plantation after application of different type of liquid biosolids (Corey et al. 1986).

Nitrogen release from biosolids was similar to inorganic fertilizer. This clearly indicated that biosolids could be a source of N to improve pine plantations productivity. A number of studies reported significant tree growth after application of biosolids to pine forest (Berry 1987; Wang et al. 2004). Prescott et al. (1993) observed no differences in tree growth response for five years following biosolids and conventional fertilizers application on *Thuja plicata*, *Tsuga heterophylla*, and *Abies amabilis*.

Our findings of differences in soil N concentration among biosolids type were similar to other studies that compared N release from biosolids (Beauchamp et al. 1979; Chae and Tabatabai 1986). N mineralized from the organic N in biosolids was 64.7% in the Pellet225, 14.6% in the LS225, and 10.1% in AD225 treatments in the 20-month period. These values were consistent with other field and laboratory studies (Cogger et al. 2004; Gilmour et al. 2003; Matsuoka et al. 2006; Parnaudeau et al. 2004). The lower N mineralization in the AD225 agrees with other studies reporting limited N mineralization in anaerobically digested biosolids (Garau et al. 1986; Wang et al. 2003).

Several processes explain differences in N availability among biosolids treatments. First, other researches attributed differences in mineralization rate due to the rapid mineralization of the labile portion of the biosolids, and the slow decomposition of the more recalcitrant organic N resistant to mineralization (Boyle and Paul 1989a; Gilmour et al. 1996; Lerch et al. 1992; Smith et al. 1998). Second, a number of studies indicated that much of the N available or mineralized following surface application of biosolids can be lost through volatilization, immobilization, and denitrification (Donovan and Logan 1983; Epstein et al. 1978). Anaerobically digested biosolids

were shown to lose 60% of N through volatilization during the five-day period following surface application (Beauchamp et al. 1978), and between 71 to 81% within a 3 weeks period when they were applied to forest (Robinson et al. 2002). The total precipitations following treatments application in March 2006 was particularly low (Fig 3.1) and favored N volatilization from biosolids.

In our study, the initial release of N observed in the biosolids during the first year was a combination of release of inorganic N and mineralization. In the AD225 and the U+DAP225, the initial pulse of available N measured in the IEM suggests release of inorganic N present at the start (Table 3.2). This release pattern was similar to the findings reported by Mudano (1986) following conventional fertilization in a loblolly pine plantation in the Piedmont. This rapid response in soil nitrogen was also observed on several biosolids studies (Boyle and Paul 1989b; Hallett et al. 1999; Terry et al. 1979; Wang et al. 2003), and it has been attributed to the initial inorganic N content in the biosolids, and the rapid mineralization of the labile organic N in biosolids.

In contrast, N released from the Pellet225 was mainly produced by mineralization of the organic N. We observed that higher N mineralization in the pellet225 corresponded to the continued elevate NH_4-N observed in the IEM at the same sampling time (Fig 3.3.a). Rapid N mineralization has been reported by Tarrason et al (2008) when they compared thermally dried with anaerobically digested biosolids, and it is explained by the high proportion of labile organic N in pelletized or thermally dried biosolids (Smith and Durham 2002; Smith et al. 1998). Eldridge et al (2008) determined that about 50% of N mineralization occurred within two months after surface application of pelletized biosolids. Kelty et al. (2004) reported that 26% of the organic N applied with pelletized biosolids was rapidly mineralized following surface

application in red pine forest.

Initial IEM-NH_4 in the LS225 treatment was much lower than the other biosolids. The lime stabilized biosolids used in this experiment was aerobically digested, which have been found to mineralized more rapid than other biosolids (Garau et al. 1986). Other studies observed that lime stabilized biosolids increased surface soil pH and N mineralization in acid soils (Little et al. 1991; Lyngstad 1992). However, the high biosolids pH are likely to increase losses of the mineralized N in biosolids through volatilization (Quemada et al. 1998; Terry et al. 1981). The initial pH in the biosolids applied in the LS225 treatment was 12.3. Donovan and Logan (1983), reported significant NH_3 losses through volatilization when pH was 7.5 in comparison to 6.7 or 5.1.

We found that IEM-NO_3 concentration increased few months after it did for the IEM-NH_4-N in the AD225, and the LS225 treatments. This lag time has been observed in other biosolids studies (Aschmann et al. 1992; Beauchamp et al. 1979), and it could be explained by the NH_4-N fixation in the clay minerals (Feigenbaum et al. 1994), the restrictive effect that high salts concentration in biosolids have on microbial activity (Aschmann et al. 1992), the effect of acidic conditions in forest floor on nitrification (Burton et al. 1990).

The higher pH and moisture content of the AD225 and the LS225 material compared to the Pellet225 may have created better conditions for microbial growth. Garau et al (1986) reported that nitrification in biosolids was greater in soils with pH 7.8 in comparison to acidic soils with pH of 5.5. Gilmour (1984) and Terry et al. (1981) also observed higher nitrification rate following biosolids application due to increases in soil pH. The higher IEM-NO_3 response in the AD225 and LS225 treatments could be explained by the effect of higher soil pH on nitrifiers following application of alkaline biosolids (Gilmour 1984; Terry et al. 1981), the nitrifiers added

with the biosolids (Burton et al. 1990), and the response to biosolids of the microbial population existing in the forest soil (Raison et al. 1992; Vitousek and Matson 1985).

Increase in surface soil NO_3-N after surface application of biosolids was reported in the field and incubation studies (Bowden and Hann 1997; Chae and Tabatabai 1986; Correa et al. 2005). They concluded that NO_3-N availability was mainly a consequence of nitrification that occurred after the biosolids application. The lag time between NH_4-N nd NO_3-N concentrations in all the treatments, was previously reported in other biosolids studies (Aschmann et al. 1992; Beauchamp et al. 1979), and it is a consequence of the low NH_4-N availability for nitrification (Boyle and Paul 1989b; Gilmour 1984), or the increase in surface soil salinity after biosolids application (Aschmann et al. 1992) that inhibited soil nitrifiers (Darrah et al. 1987; McClung and Frankenberger 1985). The effect of the acidic conditions in forest floor could also reduce nitrification from biosolids (Burton et al. 1990). The eventual increase in NO_3-N concentration observed may be a result of growing presence of nitrifiers added with the biosolids (Burton et al. 1990) or the response of the microbial population previously existing in the forest soil (Raison et al. 1992; Vitousek and Matson 1985).

All the biosolids treatments tended to decrease total N availability during winter and early in the spring and then had a second peak early during the 2007 summer. A number of studies have found that N mineralization still occurred at lower rates one or two years following biosolids application (Hallett et al. 1999; Hernandez et al. 2002; Kelty et al. 2004; Mitchell et al. 2000). The mineralization of the remaining organic N in the biosolids may have been responsible for the late response during the second year.

3.4.2. Effect of Biosolids Application Rates on N Availability

Higher application rates of anaerobically digested biosolids leaded to high IEM-N and N mineralization. However, the relationship between the application rates of biosolids and the soil N availability was not linear probably as a consequence of biosolids properties and site characteristics that affect N mineralization. Similar results have also been found with increasing application rates of biosolids in forest (Aschmann et al. 1990; Brockway and Urie 1983; Haith et al. 1992).

In this experiment, the large application of organic N increased IEM-N and N-min even after the end of the growing season and through the whole winter season. At this point, plant uptake is reduced increasing the potential for NO_3-N leaching. Aschmann et al. (1992) found that soil NO_3-N concentrations increased following the application of three increasing rates of biosolids applied to northern hardwood forest. Several studies reported deep NO_3-N leaching with concentration above safe water quality standards following application of large rates of biosolids to forest (Brockway and Urie 1983; McLaren et al. 2005; Robinson et al. 2002).

3.4.3. Effect of Season of Biosolids Application on N Availability

Season did not affect the average available N after surface application of anaerobically digested biosolids at the same rates in November 2005 (fall) and March 2006 (spring) and have little effect on monthly N-min measurements. These results could be explained by the low incorporation rate of nutrients when anaerobically digested biosolids are surface applied (Gilmour et al. 2003), the effect of soil and temperature on mineralization (Smith et al. 1998; Terry et al. 1979; Wang et al. 2003), and the effect of forest floor on nutrient mobility (Sollins et al. 1988).

3.5. Conclusions

We demonstrated that surface application of biosolids to a 17-year-old loblolly pine plantation increased soil N availability and could be use as an alternative to conventional forest fertilization. When biosolids were applied at the permitted rate of 225 PAN kg ha^{-1}, the average soil N in the pelletized biosolids was greater than the conventional fertilizer. Biosolids applied in the AD225 and the LS225 treatments have similar effect on soil N availability.

Biosolids increased soil N availability at different rates over time as a consequence of the initial inorganic N content and the N mineralization. During the first year soil N immediately increased after biosolids application, and declined by the end of the growing season. N mineralization observed during the second year provided a long-term source of soil available N like a control release fertilizer.

We found that high rates increased N availability and mineralization during the first and the second year following treatments application. Soil nitrate levels remained elevated with the 900 kg PAN ha^{-1} and 1800 PAN ha^{-1} rates during most of the study. The adequate biosolids rate based on PAN needs to maximize plant nutrient uptake, minimizing N losses. One of the major environmental concerns in the Virginia Piedmont regarding biosolids application is the potential for NO_3-N leaching and subsequent groundwater contamination.

We demonstrated that biosolids application increased N availability in loblolly pine forest. However, in order to incorporate biosolids application as a silviculture practice in the Virginia Piedmont we need to increase the understanding of how site specific characteristics and biosolids type regulated N cycling and the accumulation of other nutrients.

3.6. References

Albaugh, T.J., H.L. Allen, and T.R. Fox. 2007. Historical patterns of forest fertilization in the southeastern United States from 1969 to 2004. Southern Journal of Applied Forestry 31(3):129-137.

APHA. 1998. Standard methods for examination of water and wastewater. Am. Water Works Assoc., and Water Environment Federation, Washington, DC.

Aschmann, S.G., M.S. McIntosh, J.S. Angle, and R.L. Hill. 1992. Nitrogen movement under a hardwood forest amendd with liquid waste-water sludge. Agriculture Ecosystems & Environment 38(4):249-263.

Aschmann, S.G., M.S. McIntosh, J.S. Angle, R.L. Hill, and R.R. Weil. 1990. Nitrogen status of forest floor, soils, and vegetation following municipal waste-water sludge application. Journal of Environmental Quality 19(4):687-694.

Beauchamp, E.G., G.E. Kidd, and G. Thurtell. 1978. Ammonia volatilization from sewage sludge applied in field. Journal of Environmental Quality 7(1):141-146.

Beauchamp, E.G., Y.K. Soon, and J.R. Moyer. 1979. Nitrate Production from Chemically Treated Sewage Sludges in Soil. Journal of Environmental Quality 8(4):557-560.

Beech, N., K. Crawford, N. Goldstein, G. Kester, M. Lono-Batura, and E. Dzieyk. 2007. A national biosolids regulation, quality, end use and disposal survey: Final report. North East Biosolids and Residuals Association (NEBRA). 30.

Berry, C.R. 1987. Use of municipal sewage sludge for improvement of forest sites in the southeast. United Stated Department of Agriculture, Forest Service. 33 pp.

Bowden, W., and M.J. Hann. 1997. The availability of nitrogen following topsoil application of liquid digested sludge. Nutrient Cycling in Agroecosystems 47(2):167-172.

Boyle, M., and E.A. Paul. 1989a. Carbon and nitrogen mineralization kinetics in soil previously amended with sewage-sludge. Soil Science Society of America Journal 53(1):99-103.

Boyle, M., and E.A. Paul. 1989b. Nitrogen transformations in soils previously amended with sewage-sludge. Soil Science Society of America Journal 53(3):740-744.

Brockway, D.G. 1983. Forest Floor, Soil, and vegetation responses to sludge fertilization in Red and White-Pine Plantations. Soil Science Society of America Journal 47(4):776-784.

Brockway, D.G., and D.H. Urie. 1983. Determining Sludge Fertilization Rates for Forests from Nitrate-N in Leachate and Groundwater. Journal of Environmental Quality 12(4):487-492.

Brockway, D.G., D.H. Urie, P.V. Nguyen, and J.B. Hart. 1986. Wastewater and sludge nutrient utilization in forest ecosystems. P. 221-245 in The forest alternative for wastewater and sludge nutreatment and utilization of municipal and industrial wastes, D.W., C., C. Henry, and N. W.L. (eds.). University of Washington Press, Seattle.WA.

Burton, A.J., J.B. Hart, and D.H. Urie. 1990. Nitrification in slude-amended michigan forest soils. Journal of Environmental Quality 19(3):609-616.

Chae, Y.M., and M.A. Tabatabai. 1986. Mineralization of nitrogen in soils amended with organic wastes. Journal of Environmental Quality 15(2):193-198.

Cogger, C.G., A.I. Bary, D.M. Sullivan, and E.A. Myhre. 2004. Biosolids processing effects on first- and second-year available nitrogen. Soil Science Society of America Journal 68(1):162-167.

Cooperband, L.R., and T.J. Logan. 1994. Measuring in-Situ Changes in Labile Soil-Phosphorus with Anion-Exchange Membranes. Soil Science Society of America Journal 58(1):105-114.

Corey, J.C., M.W. Lower, and C.E. Davis. 1986. The sludge application program at the Savannah River plant. in The Forest Alternative for Treatment and Utilization of Municipal and Industrial Wastes. Proceedings of the Forest Lands Applications Symposium, Cole, D.W., C. Henry, and W.L. Nutter (eds.). University of Washington Press. Seattle, WA., Seattle, WA.

Correa, R.S., R.E. White, and A.J. Weatherley. 2005. Modelling the risk of nitrate leaching from two soils amended with five different biosolids. Revista Brasileira De Ciencia Do Solo 29(4):619-626.

Darrah, P.R., P.H. Nye, and W. R.E. 1987. The effect of high solute concentration on nitrifiction in soil. Plant and Soil 97:37-45.

Donohue, S.J., and S.E. Heckendorn. 1994. Soil test recommendations for Virginia. Virginia Tech. Blacksburg, VA.

Donovan, W.C., and T.J. Logan. 1983. Factors affecting ammonia volatilization from sewage-sludge applied to soil in a laboratory study. Journal of Environmental Quality 12(4):584-590.

Eldridge, S.M., K.Y. Chan, Z.H. Xu, C.R. Chen, and I. Barchia. 2008. Plant-available nitrogen supply from granulated biosolids: implications for land application guidelines. Australian Journal of Soil Research 46(5):423-436.

Epstein, E., D.B. Keane, J.J. Meisinger, and J.O. Legg. 1978. Mineralization of nitrogen from sewage sludge and sludge compost. Journal of Environmental Quality 7(2):217-221.

Evanylo, G.K. 1999a. Agricultural Land Application of Biosolids in Virginia: Managing Biosolids for Agricultural Use. Crop and Soil Environmental Sciences Publication. 452-303. Virginia Cooperative Extension Service.

Evanylo, G.K. 1999b. Agricultural Land Application of Biosolids in Virginia: Production and Characteristics of Biosolids. Crop and Soil Environmental Sciences Publication. 452-301. Virginia Cooperative Extension Service.

Feigenbaum, S., A. Hadas, M. Sofer, and J.A.E. Molina. 1994. Clay-fixed labeled ammonium as a source of available nitrogen. Soil Science Society of America Journal 58:980-985.

Fox, T.R., H.L. Allen, T.J. Albaugh, R. Rubilar, and C.A. Carlson. 2007. Tree nutrition and forest fertilization of pine plantations in the southern United States. Southern Journal of Applied Forestry 31(1):5-11.

Garau, M.A., M.T. Felipo, and M.C.R. Devilla. 1986. Nitrogen Mineralization of Sewage Sludges in Soils. Journal of Environmental Quality 15(3):225-228.

Gilmour, J.T. 1984. The effect of soil properties on nitrification and nitrification inhibition. Soil Science Society of America Journal 48(6):1262-1266.

Gilmour, J.T., M.D. Clark, and S.M. Daniel. 1996. Predicting long-term decomposition of biosolids with a seven-day test. Journal of Environmental Quality 25(4):766-770.

Gilmour, J.T., C.G. Cogger, L.W. Jacobs, G.K. Evanylo, and D.M. Sullivan. 2003. Decomposition and plant-available nitrogen in biosolids: Laboratory studies, field studies, and computer simulation. Journal of Environmental Quality 32(4):1498-1507.

Gove, L., F.A. Nicholson, H.F. Cook, and A.J. Beck. 2002. Comparison of the effect of surface application and subsurface incorporation of enhanced treated biosolids on the leaching of heavy metals and nutrients through sand and sandy loam soils. Environmental Technology 23(2):189-198.

Gurlevik, N., D.L. Kelting, and H.L. Allen. 2004. Nitrogen mineralization following vegetation control and fertilization in a 14-year-old loblolly pine plantation. Soil Science Society of America Journal 68(1):272-281.

Haith, D.A., J.E. Reynolds, P.T. Landre, and T.L. Richard. 1992. Sludge loading rates for forest land. Journal of Environmental Engineering-Asce 118(2):196-208.

Hallett, R.A., W.B. Bowden, and C.T. Smith. 1999. Nitrogen dynamics in forest soils after municipal sludge additions. Water Air and Soil Pollution 112(3-4):259-278.

Henry, C., D. Sullivan, R. Rynk, K. Dorsey, and C. Cogger. 1999. Managing nitrogen from biosolids. Washington State Dept. of Ecology, [Olympia? Wash.]. 75 p.

Henry, C.L., D.W. Cole, and R.B. Harrison. 1994. Use of municipal sludge to restore and improve site productivity in forestry - The pack forest sludge research program. P. 137-149 in IEA/BE Workshop on Ameliorative Practices for Restoring and Maintaining Long-Term Productivity in Forests, Vaxjo, Sweden.

Hernandez, T., R. Moral, A. Perez-Espinosa, J. Moreno-Caselles, M.D. Perez-Murcia, and C. Garcia. 2002. Nitrogen mineralisation potential in calcareous soils amended with sewage sludge. Bioresource Technology 83(3):213-219.

Jaynes, W.F., R.E. Zartman, R.E. Sosebee, and D.B. Wester. 2003. Biosolids decomposition after surface applications in west Texas. Journal of Environmental Quality 32(5):1773-1781.

Jokela, E.J., W.H. Smith, and S.R. Colbert. 1990. Growth and elemental content of slash pine 16 years after treatment with garbage composted with sewage-sludge. Journal of Environmental Quality 19(1):146-150.

Kane, P.F. 2000. AOAC Methods. Association of Official Analytical Chemists.Washington, DC.

Kelty, M.J., F.D. Menalled, and M.M. Carlton. 2004. Nitrogen dynamics and red pine growth following application of pelletized biosolids in Massachusetts, USA. Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 34(7):1477-1487.

Lerch, R.N., K.A. Barbarick, L.E. Sommers, and D.G. Westfall. 1992. Sewage-Sludge Proteins as Labile Carbon and Nitrogen-Sources. Soil Science Society of America Journal 56(5):1470-1476.

Littell, R., G. Milliken, W. Stroup, R. Wolfinger, and O. Schabenberger. 2006. SAS for Mixed Models, Second Edition. SAS Press, Cary, NC.

Little, D.A., R.B. Reneau, and D.C. Martens. 1991. Lime-stabilized and chemical-fixed sewage sludges as lime amendments. Bioresource Technology 37(1):93-102.

Lyngstad, I. 1992. Effect of Liming on Mineralization of Soil-Nitrogen as Measured by Plant Uptake and Nitrogen Released during Incubation. Plant and Soil 144(2):247-253.

Magesan, G.N., C.D.A. McLay, and V.V. Lal. 1998. Nitrate leaching from a free-draining volcanic soil irrigated with municipal sewage effluent in New Zealand. Agriculture Ecosystems & Environment 70(2-3):181-187.

Matsuoka, K., N. Moritsuka, T. Masunaga, K. Matsui, and T. Wakatsuki. 2006. Effect of heating treatments on nitrogen mineralization from sewage sludge. Soil Science and Plant Nutrition 52(4):519-527.

McClung, G., and W.T. Frankenberger. 1985. Soil nitrogen transformatins as affected by salinity. Soil Sci 139:404-411.

McLaren, R.G., L.M. Clucas, T.W. Speir, and A.P. van Schaik. 2007. Distribution and movement of nutrients and metals in a Pinus radiata forest soil following applications of biosolids. Environmental Pollution 147(1):32-40.

McLaren, R.G., L.M. Clucas, and M.D. Taylor. 2005. Leaching of macronutrients and metals from undisturbed soils treated with metal-spiked sewage sludge. 3. Distribution of residual metals. Australian Journal of Soil Research 43(2):159-170.

Medalie, L., W.B. Bowden, and C.T. Smith. 1994. Nutrient Leaching Following Land Application of Aerobically Digested Municipal Sewage-Sludge in a Northern Hardwood Forest. Journal of Environmental Quality 23(1):130-138.

Mehlich, A. 1953. Determination of P, Ca, Mg, K, Na, NH4. North Carolina Dept. of Agriculture, Agronomic Division.

Mitchell, D.S., A.C. Edwards, and R.C. Ferrier. 2000. Changes in fluxes of N and P in water draining a stand of Scots pine treated with sewage sludge. Forest Ecology and Management 139(1-3):203-213.

Mudano, J.E. 1986. Assessment of Soil Nitrogen Availability Following Nitrogen and Phosphorus Fertilization of a Loblolly Pine Stand, North Carolina State University, Raleigh, NC. 52 p.

N.R.C. 2002. Biosolids applied to land : advancing standards and practices. National Academies Press. . xviii, 345 p.

Parnaudeau, V., B. Nicolardot, and J. Pages. 2004. Relevance of organic matter fractions as predictors of wastewater sludge mineralization in soil. Journal of Environmental Quality 33(5):1885-1894.

Parnaudeau, V., B. Nicolardot, P. Robert, G. Alavoine, J. Pages, and F. Duchiron. 2006. Organic matter characteristics of food processing industry wastewaters affecting their C and N mineralization in soil incubation. Bioresource Technology 97(11):1284-1295.

Powers, R.F. 1980. Mineralizable soil-nitrogen as an index of nitrogen availability to forest trees. Soil Science Society of America Journal 44(6):1314-1320.

Prescott, C.E., and L.L. Blevins. 2005. Eleven-year growth response of young conifers to biosolids or nitrogen and phosphorus fertilizer on northern Vancouver Island. Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 35(1):211-214.

Prescott, C.E., M.A. McDonald, S.P. Gessel, and J.P. Kimmins. 1993. Long-term effects of sewage-sludge and inorganic fertilizers on nutrient turnover in litter in a coastal Douglas-fir forest. Forest Ecology and Management 59(1-2):149-164.

Prescott, C.E., and L.M. Zabek. 1997. Growth response and nutrient availability in western redcedar plantations following amendment with fish-wood compost and straw. Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 27(4):598-602.

Quemada, M., B. Lassa, C. Lamsfus, and P.M. Aparicio Tejo. 1998. Ammonia Volatilization from Surface or Incorporated Biosolids by the Addition of Dicyandiamide. Journal of Environmental Quality 27:980-983.

Raison, R.J., M.J. Connell, and P.K. Khanna. 1987. Methodology for Studying Fluxes of Soil Mineral-N Insitu. Soil Biology & Biochemistry 19(5):521-530.

Raison, R.J., M.J. Connell, P.K. Khanna, and R.A. Falkiner. 1992. Effects of Irrigation and Nitrogen-Fertilization on Fluxes of Soil Mineral Nitrogen in a Stand of Pinus-Radiata. Forest Ecology and Management 52(1-4):43-64.

Robinson, M.B., and P.J. Polglase. 2000. Volatilization of nitrogen from dewatered biosolids. Journal of Environmental Quality 29(4):1351-1355.

Robinson, M.B., P.J. Polglase, and C.J. Weston. 2002. Loss of mass and nitrogen from biosolids applied to a pine plantation. Australian Journal of Soil Research 40(6):1027-1039.

Rowell, D.M., C.E. Prescott, and C.M. Preston. 2001. Decomposition and nitrogen mineralization from biosolids and other organic materials: Relationship with initial chemistry. Journal of Environmental Quality 30(4):1401-1410.

Simmons, J.A., J.B. Yavitt, and T.J. Fahey. 1996. Watershed liming effects on the forest floor N cycle. Biogeochemistry 32(3):221-244.

Smith, S.R., and E. Durham. 2002. Nitrogen release and fertiliser value of thermally-dried biosolids. Journal of the Chartered Institution of Water and Environmental Management 16(2):121-126.

Smith, S.R., V. Woods, and T.D. Evans. 1998. Nitrate dynamics in biosolids-treated soils. I. Influence of biosolids type and soil type. Bioresource Technology 66:139-149.

Sollins, P., G.P. Robertson, and G. Uehara. 1988. Nutrient mobility in variable and permanent-charge soils. Biogeochemistry 6:181-199.

Subler, S., J.M. Blair, and C.A. Edwards. 1995. Using Anion-Exchange Membranes to Measure Soil Nitrate Availability and Net Nitrification. Soil Biology & Biochemistry 27(7):911-917.

Tarrason, D., G. Ojeda, O. Ortiz, and J.M. Alcaniz. 2008. Differences on nitrogen availability in a soil amended with fresh, composted and thermally-dried sewage sludge. Bioresource Technology 99(2):252-259.

Terry, R.E., D.W. Nelson, and L.E. Sommers. 1979. Decomposition of anaerobically digested sewage sludge as affected by soil environmental-conditions. Journal of Environmental Quality 8(3):342-347.

Terry, R.E., D.W. Nelson, and L.E. Sommers. 1981. Nitrogen transformation in sewage sludge amended soils as affected by soil environmental factors. Soil Science Society of America Journal 45(3):506-513.

USEPA. 1983. Methods for the chemical analysis of water and wastes (MCAWW). EPA/600/4-79/020. NTIS item PB84-128677. in Environ. Monitoring and Support Lab. Office of Res. and Dev., USEPA, Cincinnati, OH.

USEPA. 1995. Process design for agricultural utilization. In Process Design Manual–Land Application of Sewage Sludge and Domestic Septage EPA/625/R-95/001; U.S. Environmental protection agency. Washington, DC.

USEPA. 2002. Test methods for evualating solid waste, physical/chemical methods SW-846 manual U.S. Environmental protection Agency. U.S. Gov Print Office.

VDH. 2004. Report of the Virginia Department of Health on the U.S. Environmental Protection Agency's response to the National Research Council's report pertaining to the land application of biosolids to the Governor and the General Assembly of Virginia. Commonwealth of Virginia, Richmond, Va. 20 pp.

Vitousek, P.M., and P.A. Matson. 1985. Disturbance, nitrogen availability, and nitrogen losses in a intensively managed loblolly pine plantation. Ecology 66:1360-1376.

Wang, H.L., M.O. Kimberley, and M. Schlegelmilch. 2003. Biosolids-derived nitrogen mineralization and transformation in forest soils. Journal of Environmental Quality 32(5):1851-1856.

Wang, H.L., G.N. Magesan, M.O. Kimberley, T.W. Payn, P.J. Wilks, and C.R. Fisher. 2004. Environmental and nutritional responses of a Pinus radiata plantation to biosolids application. Plant and Soil 267(1-2):255-262.

3.7. Tables and Figures

Table 3.1. Selected chemical and physical properties from the upper mineral soil from a 17-year-old loblolly pine plantation, Amelia County, VA.

Soil Depth (cm)	PH (1:1)	BD g cc^{-1}	C	N	P	K	Ca	Mg
			mg Kg^{-1}					
0-20	5.47	1.24	5700	484.0	4.0	32.0	342.0	73.0

Table 3.2. Selected properties for biosolids surface applied in a 17-year-old loblolly pine plantation in Amelia County, VA. Biosolids source for the fall application treatment was Alexandria (VA). Biosolids sources for the spring application were Blue Plain (DC) and Back River (MD), and Baltimore (MD).

Properties	Lime stabalized (Blue Plain)	Anaerobically Digested (Alexandria)	Anaerobically Digested (Back River)	Pelletized (Baltimore)
pH	12.3	8.1	8.2	5.6
		mg kg^{-1}		
Solids	352300	241500	205100	929500
Nitrogen (TKN)	31300	47500	50300	56600
Water Insol N	-	-	-	50900
Ammonia-N	1100	10200	11800	600
Phosphorus	10600	17300	20200	16100
Potassium	1300	1100	2100	2700
Sulfur	4300	10400	9300	5500
Calcium	114300	29400	22400	11200
Magnesium	2300	3300	3800	2200
Sodium	200	500	1000	400
Iron	44693	50749	55100	13682
Manganese	197	946	793	159
Copper	158	403	463	261
Zinc	314	796	867	395
Cadmium	1.2	5.8	10	-
Chromium	38	76	75	89
Nickel	16	37	36	14
Lead	38	66	66	20
Arsenic	2.9	3.6	2.1	3.9
Mercury	0.3	1.4	1.04	0.4
Selenium	2.0	3.6	4.7	2.1

Treatments	Treatment name	Target PAN	Actual PAN	Total N	N-Org	Total P	Total K	Total Ca	Dry Weight
					Kg ha^{-1}				Mg ha^{-1}
Fall Application									
Anaer. Digested	AD900F	900	847	3202	2514	1166	74	1982	67.4
Spring Application									
Lime Stabilized	LS225	225	275	920	888	312	38	3360	29.4
Pellets	Pellet225	225	306	306	291	84	14	58	5.2
Urea + DAP	U+DAP225	225	214	214	0	23	-	-	-
Anaer. Digested	AD225	225	306	1132	866	455	47	504	22.5
Anaer. Digested	AD900S	900	860	3179	2433	1277	133	1416	63.2
Anaer. Digested	AD1800	1800	1786	6604	5055	2652	276	2941	131.3

Table 3.3. Final application rates and nutrient applied after of surface application of biosolids in a 17-year-old loblolly pine plantation in Amelia County, VA. Fall treatment application was anaerobically digested biosolids at a rate of 900 kg PAN ha^{-1}. Spring treatments applications were lime stabilized, pelletized, and anaerobically digested biosolids, and urea+DAP fertilizer applied at a rate of 225 kg PAN ha^{-1}, and control. Anaerobically digested biosolids was also added at a rate of 900 kg PAN ha^{-1} and 1800 kg PAN ha^{-1}.

Table 3.4. Summary of Anova for the effect of biosolids and sampling time on IEM-NH_4, IEM-NO_3, IEM-N_t, and N-mineralization following surface application of biosolids and conventional fertilizer in a 17-year-old loblolly pine plantation in Amelia County, VA.

		IEM-NH_4		IEM-NO_3		IEM-N_t		N-Min	
Source	**Num DF**	**F Value**	**Pr > F**	**F Value**	**Pr > F**	**F Value**	**Pr > F**	**F Value**	**Pr > F**
Block	3	0.62	0.6034	7.12	0.0002	2.07	0.1063	0.14	0.9373
Treatment	7	68.65	<.0001	138.22	<.0001	173.05	<.0001	41.95	<.0001
time	19	27.27	<.0001	14.85	<.0001	38.06	<.0001	25.34	<.0001
Treatment*time	133	2.46	<.0001	3.83	<.0001	3.3	<.0001	5.25	<.0001

Table 3.5. Contrast for IEM-NH_4, IEM-NO_3, IEM-Nt, and N-Mineralization from the forest floor and surface mineral soil means averaged over 20 months in a 17-year-old loblolly pine plantation following surface application of biosolids. Treatments were control, anaerobically digested biosolids (AD225, AD900F, AD900, AD1800), lime stabilized biosolids (LS225), pelletized biosolids (Pellet225), and Urea+DAP225.

Contrast	IEM-NH_4	IEM-NO_3	IEM-N_t	N-Min
		Pr > F		
Type of biosolids				
Control vs LS225	<.0001	<.0001	<.0001	<.0001
Control vs AD225	<.0001	<.0001	<.0001	0.0002
Control vs Pellet225	<.0001	<.0001	<.0001	<.0001
Control vs U+DAP225	<.0001	<.0001	<.0001	0.0037
AD225 vs U+DAP225	0.0003	<.0001	0.0913	0.4743
LS225 vs AD225	0.7234	0.1483	0.7482	0.2486
LS225 vs Pellet225	<.0001	<.0001	0.01	0.2749
LS225 vs U+DAP225	0.0009	<.0001	0.045	0.0678
Pellet225 vs AD225	<.0001	0.0009	0.0039	0.0253
Pellet225 vs U+DAP225	0.0303	0.0022	<.0001	0.0042
Application rate				
AD1800 vs AD900	<.0001	0.0004	<.0001	0.0634
AD1800 vs AD225	<.0001	<.0001	<.0001	<.0001
AD900 vs AD225	<.0001	<.0001	<.0001	<.0001
Season of application				
AD900F vs AD900S	0.0974	0.1185	0.0945	0.0427

Table 3.6. Estimated IEM-NH_4 means at each sampling date following surface application of biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County VA at the rate of 225 kg PAN ha^{-1}. Treatments were control, anaerobically digested (AD225), lime stabilized (LS225), and pelletized (Pellet225) biosolids, and urea + DAP225. Units are mg of NH_4 per m^2 of IEM surface per day. Different letters at each month indicate significant difference between treatments ($\alpha<0.05$).

Month	Control	AD225	LS225	Pellet225	U+DAP 225
			IEM-NH_4		
Feb-06	0.66	0.62	0.62	0.47	0.78
Mar-06	0.75 c	8.70 a	2.19 b	13.94 a	13.76 a
Apr-06	1.09 b	7.98 a	2.45 b	15.54 a	12.57 a
May-06	0.80 b	9.56 a	2.72 b	10.87 a	15.86 a
Jun-06	0.54 c	8.33 a	1.92 b	10.75 a	9.21 a
Jul-06	1.01 c	3.10 bc	6.54 b	23.11 a	5.77 b
Aug-06	0.59 c	0.85 c	1.99 bc	16.71 a	3.43 b
Sep-06	0.50 c	0.92 c	4.95 b	15.81 a	3.29 b
Oct-06	1.43 b	1.00 b	6.32 ab	6.52 a	1.68 b
Nov-06	1.03 ab	1.29 ab	0.66 b	2.91 a	1.85 a
Dec-06	0.59 b	1.10 b	1.32 ab	3.06 a	1.87 ab
Jan-07	0.55	1.14	0.72	2.25	1.04
Feb-07	0.43 b	0.83 ab	0.46 ab	1.72 a	0.76 ab
Mar-07	0.99	1.42	1.66	1.77	1.24
Apr-07	1.54	1.22	3.48	2.47	1.95
May-07	0.73	0.98	2.46	0.88	1.51
Jun-07	0.52 b	3.29 a	2.47 ab	1.75 a	2.29 a
Jul-07	0.56 c	1.34 bc	1.59 ab	3.39 a	2.98 a
Aug-07	0.56 b	1.21 ab	1.23 ab	2.06 ab	2.24 a
Sep-07	0.90	0.98	1.05	0.71	1.60

Table 3.7. Estimated IEM-NO_3 means at each sampling date following surface application of biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County VA, at the equivalent rates of 225 kg PAN ha^{-1}. Treatments were control, anaerobically digested (AD225), lime stabilized (LS225), and pelletized (Pellet225) biosolids, and Urea+DAP225. Units are mg of NO_3-N per m^2 of IEM surface per day. Different letters at each month indicate significant difference between treatments ($\alpha<0.05$).

Month	Control	AD225	LS225	Pellet225	U+DAP225
			IEM-NO_3		
Feb-06	0.29	0.40	0.31	0.18	0.12
Mar-06	0.43 b	2.36 a	2.32 a	1.94 a	1.26 ab
Apr-06	0.17 c	2.34 ab	3.17 a	1.42 b	1.97 ab
May-06	0.34 b	2.78 a	4.35 a	2.48 a	3.42 a
Jun-06	0.40 c	3.42 ab	6.93 a	0.97 b	1.97 ab
Jul-06	0.12 c	5.68 a	5.94 a	1.02 b	1.79 ab
Aug-06	0.40 b	8.69 a	14.68 a	1.67 b	0.77 b
Sep-06	0.08 c	8.61 a	1.85 b	0.82 b	0.11 c
Oct-06	0.09 c	7.60 a	3.94 a	1.07 b	0.12 c
Nov-06	0.17 c	0.97 bc	2.42 a	2.18 ab	0.43 c
Dec-06	0.11 c	0.60 ab	0.15 c	0.84 a	0.18 bc
Jan-07	0.23 b	0.12 c	0.88 a	0.52 ab	0.31 ab
Feb-07	0.09 ab	0.03 c	0.16 ab	0.50 a	0.04 bc
Mar-07	0.30	1.39	2.73	1.47	0.36
Apr-07	0.37 ab	1.05 a	1.35 a	1.22 a	0.24 b
May-07	0.44 c	2.60 ab	5.11 a	1.57 abc	0.85 bc
Jun-07	0.19 b	2.11 a	4.12 a	0.76 ab	0.39 b
Jul-07	0.16 c	2.11 a	2.19 a	0.46 bc	0.63 ab
Aug-07	0.41	1.47	0.96	1.31	0.91
Sep-07	0.60	0.80	1.36	1.14	0.55

Table 3.8. Estimated IEM-Nt means at each sampling date following surface application of biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County, VA, at the equivalent rates of 225 kg PAN ha^{-1}. Treatments were control, anaerobically digested (AD225), lime stabilized (LS225), and pelletized (Pellet225) biosolids, and Urea+DAP225. Units are mg of N (IEM-NH_4 + IEM-NO_3) per m^2 of IEM surface per day. Different letters at each month indicate significant difference between treatments (α<0.05).

Month	Control	AD225	LS225	Pellet225	U+DAP225
			IEM-Nt		
Feb-06	0.95	1.02	0.93	0.65	0.90
Mar-06	1.19 b	11.06 a	4.51 ab	15.87 a	15.01 a
Apr-06	1.26 c	10.32 ab	5.62 b	16.97 a	14.54 ab
May-06	1.14 b	12.33 a	7.08 ab	13.35 a	19.27 a
Jun-06	0.94 c	11.75 ab	8.84 b	11.72 a	11.18 ab
Jul-06	1.12 c	8.78 b	12.48 b	24.13 a	7.56 b
Aug-06	0.99 c	9.53 b	16.67 ab	18.38 a	4.20 c
Sep-06	0.57 d	9.53 b	6.80 c	16.63 a	3.40 d
Oct-06	1.52 c	8.59 a	10.26 a	7.60 b	1.81 c
Nov-06	1.20 b	2.25 b	3.09 a	5.10 a	2.27 b
Dec-06	0.70 c	1.70 b	1.47 c	3.89 a	2.05 b
Jan-07	0.78 b	1.26 b	1.60 ab	2.76 a	1.35 b
Feb-07	0.52 b	0.86 a	0.63 ab	2.21 a	0.80 ab
Mar-07	1.29 b	2.81 a	4.39 a	3.23 a	1.60 ab
Apr-07	1.91 b	2.27 ab	4.84 a	3.70 a	2.19 b
May-07	1.18 c	3.58 ab	7.57 a	2.45 abc	2.36 bc
Jun-07	0.71 b	5.40 a	6.59 a	2.51 b	2.68 b
Jul-07	0.72 b	3.45 a	3.78 a	3.85 a	3.61 a
Aug-07	0.97	2.68	2.20	3.37	3.15
Sep-07	1.50	1.77	2.41	1.85	2.16

Table 3.9. Estimated N-mineralization rate means at each sampling date after surface application of biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County VA at the equivalent rate of 225 kg PAN ha^{-1}. Treatments were control, anaerobically digested (AD225), lime stabilized (LS225), and pelletized (Pellet225) biosolids, and urea + DAP225. Different letters at each month indicate significant difference between treatments (α<0.05).

Month	Control	AD 225	LS 225	Pellet225	U+DAP 225
	N-Min (kg ha^{-1})				
Feb-06	5.5	3.9	4.9	6.8	4.9
Mar-06	7.74	7.08	6.79	5.20	9.4
Apr-06	3.10 b	9.44 ab	5.35 b	12.43 ab	16.2 a
May-06	14.31 ab	11.72 ab	6.87 b	22.80 ab	42.0 a
Jun-06	14.79	16.96	16.50	26.39	48.1
Jul-06	5.63 b	11.74 b	15.21 ab	28.13 ab	68.9 a
Aug-06	4.08 b	10.87 ab	23.26 a	38.34 a	25.1 a
Sep-06	2.24 b	13.02 a	22.00 a	33.07 a	12.4 a
Oct-06	3.21 b	14.29 a	22.60 a	18.76 a	8.9 ab
Nov-06	10.12 ab	5.26 bc	13.91 a	10.70 ab	3.3 c
Dec-06	2.73 ab	1.87 b	11.49 a	6.99 a	3.0 ab
Jan-07	4.41	4.77	5.19	1.79	3.6
Feb-07	2.51 ab	6.74 a	3.66 a	1.37 b	1.3 b
Mar-07	4.41 ab	11.95 a	2.82 b	8.19 a	5.4 a
Apr-07	13.87	17.30	18.44	14.99	7.5
May-07	4.15 b	15.47 ab	26.70 a	10.34 ab	4.8 ab
Jun-07	6.80 ab	37.08 a	16.98 ab	21.23 ab	6.4 b
Jul-07	18.09	9.82	13.57	33.50	9.1
Aug-07	9.87	8.12	16.68	16.47	6.2
Sep-07	5.63	13.55	20.19	13.96	11.9

Table 3.10. Estimated IEM-NH_4 means at each sampling date after surface application of biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County VA. Treatments rates were 225, 900, and 1800 kg PAN ha^{-1} of anaerobically digested biosolids. Units are mg of NH_4 per m^2 of IEM surface per day. Different letters at each month indicate significant difference between treatments (α<0.05).

Month	AD225	AD900	AD1800
		IEM-NH_4	
Feb-06	0.62	0.63	0.69
Mar-06	8.70 b	16.68 b	43.68 a
Apr-06	7.98 b	25.20 ab	33.11 a
May-06	9.56	15.33	31.35
Jun-06	8.33	13.69	22.51
Jul-06	3.10 b	10.82 a	13.64 a
Aug-06	0.85 b	7.40 a	10.78 a
Sep-06	0.92 b	3.65 a	8.41 a
Oct-06	1.00 b	6.62 a	9.18 a
Nov-06	1.29 b	6.58 a	10.40 a
Dec-06	1.10 b	8.20 a	3.07 a
Jan-07	1.14 b	7.61 a	6.69 a
Feb-07	0.83 b	3.68 a	4.45 a
Mar-07	1.42 b	6.68 a	4.78 a
Apr-07	1.22 b	2.53 b	9.34 a
May-07	0.98 b	2.94 ab	4.42 a
Jun-07	3.29	3.24	5.58
Jul-07	1.34 b	2.01 ab	4.31 a
Aug-07	1.21	1.57	6.38
Sep-07	0.98 b	1.72 b	2.72 a

Table 3.11. Estimated IEM-NO_3 means at each sampling date after surface application of biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County VA. Treatments rates were 225, 900, and 1800 kg PAN ha^{-1} of anaerobically digested biosolids. Units are mg of NO_3 per m^2 of IEM surface per day. Different letters at each month indicate significant difference between treatments (α<0.05).

Month	AD225	AD900	AD1800
		IEM-NO_3	
Feb-06	0.40	0.44	0.07
Mar-06	2.36 ab	0.97 b	4.04 a
Apr-06	2.34	3.19	8.23
May-06	2.78 b	12.57 a	10.71 a
Jun-06	3.42 b	8.56 ab	17.32 a
Jul-06	5.68	12.52	18.44
Aug-06	8.69	16.99	25.76
Sep-06	8.61	12.33	32.84
Oct-06	7.60	7.73	12.65
Nov-06	0.97 b	8.33 a	12.15 a
Dec-06	0.60 c	15.34 a	2.22 b
Jan-07	0.12 b	2.41 a	2.39 a
Feb-07	0.03 c	9.95 a	1.70 b
Mar-07	1.39 b	7.14 a	5.48 a
Apr-07	1.05 b	10.16 a	9.27 a
May-07	2.60	3.47	3.96
Jun-07	2.11	5.65	8.47
Jul-07	2.11	1.95	5.11
Aug-07	1.47	1.83	6.28
Sep-07	0.80 b	1.58 b	7.05 a

Table 3.12. Estimated IEM-Nt means at each sampling date after surface application of biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County VA. Treatments rates were 225, 900, and 1800 kg PAN ha^{-1} of anaerobically digested biosolids. Units are mg of N (IEM-NH_4 + IEM-NO_3) per m^2 of IEM surface per day. Different letters at each month indicate significant difference between treatments (α<0.05).

Month	AD225	AD900	AD1800
		IEM-Nt	
Feb-06	1.02	1.07	0.76
Mar-06	11.06 b	17.66 b	47.71 a
Apr-06	10.32	28.38	41.34
May-06	12.33 b	27.90 a	42.06 a
Jun-06	11.75 b	22.25 ab	39.83 a
Jul-06	8.78	23.34	32.09
Aug-06	9.53	24.39	36.54
Sep-06	9.53	15.98	41.25
Oct-06	8.59	14.35	21.83
Nov-06	2.25 b	14.90 a	22.55 a
Dec-06	1.70 c	23.54 a	5.30 b
Jan-07	1.26 b	10.02 a	9.08 a
Feb-07	0.86 c	13.63 a	6.16 b
Mar-07	2.81 b	13.81 a	10.25 a
Apr-07	2.27 b	12.69 a	18.61 a
May-07	3.58	6.41	8.38
Jun-07	5.40	8.88	14.05
Jul-07	3.45	3.96	9.42
Aug-07	2.68	3.40	12.66
Sep-07	1.77 b	3.30 b	9.77 a

Table 3.13. Estimated N-Mineralization means at each sampling date after surface application of biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County, VA. Application rates were 225, 900, and 1800 kg PAN ha^{-1} of anaerobically digested biosolids. Different letters at each month indicate significant difference (α<0.05).

Month	AD 225	AD 900	AD 1800
	N-Min (kg ha^{-1})		
Feb-06	3.9	4.7	2.8
Mar-06	7.1 b	17.6 b	59.8 a
Apr-06	9.4 b	19.8 b	81.8 a
May-06	11.7 b	49.9 a	134.0 a
Jun-06	17.0 b	101.4 a	157.8 a
Jul-06	11.7 b	161.5 a	75.2 a
Aug-06	10.9 b	61.5 a	141.0 a
Sep-06	13.0 b	117.3 a	173.2 a
Oct-06	14.3 b	82.1 a	77.6 a
Nov-06	5.3 b	58.1 a	23.1 b
Dec-06	1.9 b	29.8 a	1.1 b
Jan-07	1.9 b	9.4 a	2.4 b
Feb-07	6.7 b	1.9 b	9.2 a
Mar-07	11.9 ab	5.1 b	16.4 a
Apr-07	17.3 ab	7.8 b	39.5 a
May-07	15.5 b	37.0 ab	95.7 a
Jun-07	37.1	32.5	39.5
Jul-07	9.8 b	55.8 a	27.5 ab
Aug-07	8.1	20.0	16.0
Sep-07	13.6	18.4	22.2

Table 3.14. Estimated IEM-NH_4 means at each sampling date after surface application of biosolids on November 2005, and March 2006 to a 17-year-old loblolly pine plantation in Amelia County VA. Treatment rate was 900 kg PAN ha^{-1} of anaerobically digested biosolids. Units are mg of NH_4 per m^2 of IEM surface per day. Different letter at each month indicate significant difference (α<0.05).

Month since Application	AD 900F	AD 900S
	IEM-NH_4	
1	21.97 a	0.63 b
2	27.93	16.68
3	15.57	25.20
4	9.22	15.33
5	16.98	13.69
6	11.43	10.82
7	21.02 a	7.40 b
8	9.71	3.65
9	5.12	6.62
10	6.99	6.58
11	3.27	8.20
12	3.59	1.11
13	2.36	3.68
14	2.30 b	6.68 a
15	0.94 b	2.53 a
16	1.36 b	2.94 a
17	2.16	3.24
18	1.46	2.01
19	2.33	1.57
20	1.23	1.72

Table 3.15. Estimated IEM-NO_3 means at each sampling date after surface application of biosolids on November 2005, and March 2006 to a 17-year-old loblolly pine plantation in Amelia County VA. Treatments rate was 900 kg PAN ha^{-1} of anaerobically digested biosolids. Units are mg of NO_3 per m^2 of IEM surface per day. Different letters at each month indicate significant difference (α<0.05)

Month since Application	AD 900F	AD900S
	IEM-NO_3	
1	1.49	0.44
2	2.51	0.97
3	1.29	3.19
4	2.30 b	12.57 a
5	2.27 b	8.56 a
6	5.73	12.52
7	10.74	16.99
8	14.20	12.33
9	22.31	7.73
10	7.95	8.33
11	7.99	15.34
12	4.06	1.52
13	1.44 b	9.95 a
14	4.16	7.14
15	1.82 b	10.16 a
16	4.25	3.47
17	1.59	5.65
18	3.21	1.95
19	3.81	1.83
20	2.73	1.58

Table 3.16. Estimated IEM-Nt means at each sampling date after surface application of biosolids on November 2005, and March 2006 to a 17-year-old loblolly pine plantation in Amelia County VA. Treatments rates were 900 kg PAN ha^{-1} of anaerobically digested biosolids. Units are mg of N ($IEM\text{-}NH_4$ + $IEM\text{-}NO_3$) per m^2 of IEM surface per day. Different letters at each month indicate significant difference (α<0.05).

Month since Application	AD 900F	AD 900S
	IEM-Nt	
1	23.46	1.07
2	30.44	17.66
3	16.86	28.38
4	11.52 b	27.90 a
5	19.24 b	22.25 a
6	17.16	23.34
7	31.75	24.39
8	23.91	15.98
9	27.43	14.35
10	14.94	14.90
11	11.25	23.54
12	7.65	2.63
13	3.79 b	13.63 a
14	6.46	13.81
15	2.76 b	12.69 a
16	5.61	6.41
17	3.74	8.88
18	4.68	3.96
19	6.14	3.40
20	3.96	3.30

Table 3.17. Estimated means since application for N mineralization after surface application of anaerobically digested biosolids on November 2005 (fall), and March 2006 (spring) to a 17-year-old loblolly pine plantation in Amelia County, VA. Application rates were 900 kg PAN ha^{-1} . Different letters at each month indicate significant difference between treatments (α<0.05).

Month since Application	AD900F	AD900S
	N-Min (kg ha^{-1})	
1	3.30	4.70
2	15.3	17.65
3	5.5	19.77
4	11.9 b	49.87 a
5	66.1	101.41
6	70.6	161.47
7	108.9	61.53
8	129.9	117.28
9	62.5	82.12
10	38.0	58.12
11	57.0	29.77
12	38.6 a	9.43 b
13	15.1 a	1.85 b
14	3.8	5.12
15	3.0	7.78
16	2.7 b	37.00 a
17	3.7 b	32.54 a
18	22.0	55.77
19	7.6	20.04
20	23.0	18.43

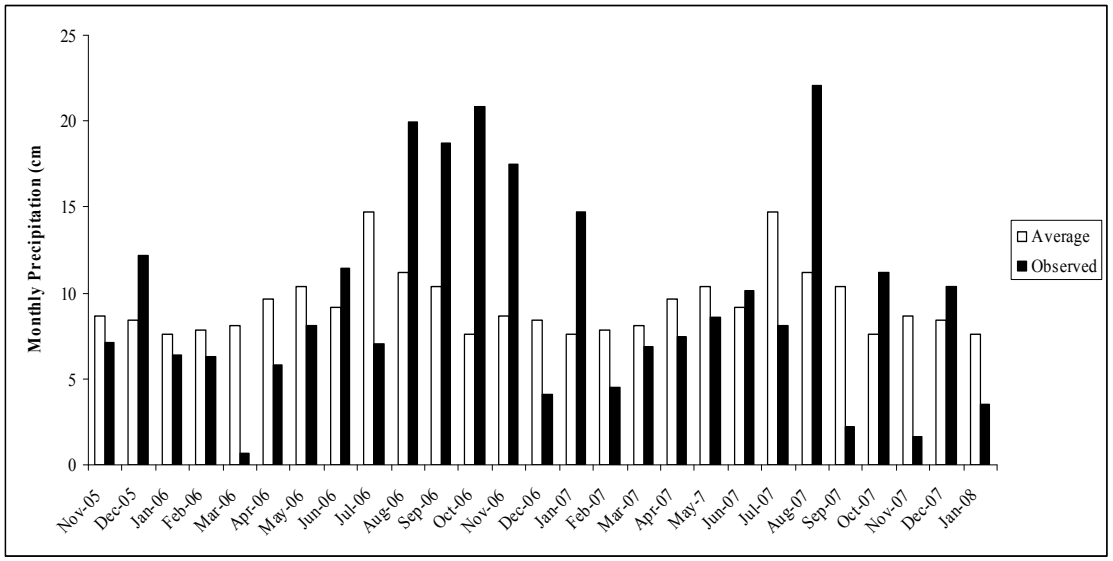


Figure 3.1. Average precipitation from 15-year period, (Blackstone, VA) and observed total monthly precipitation during the study duration (Amelia County, VA).

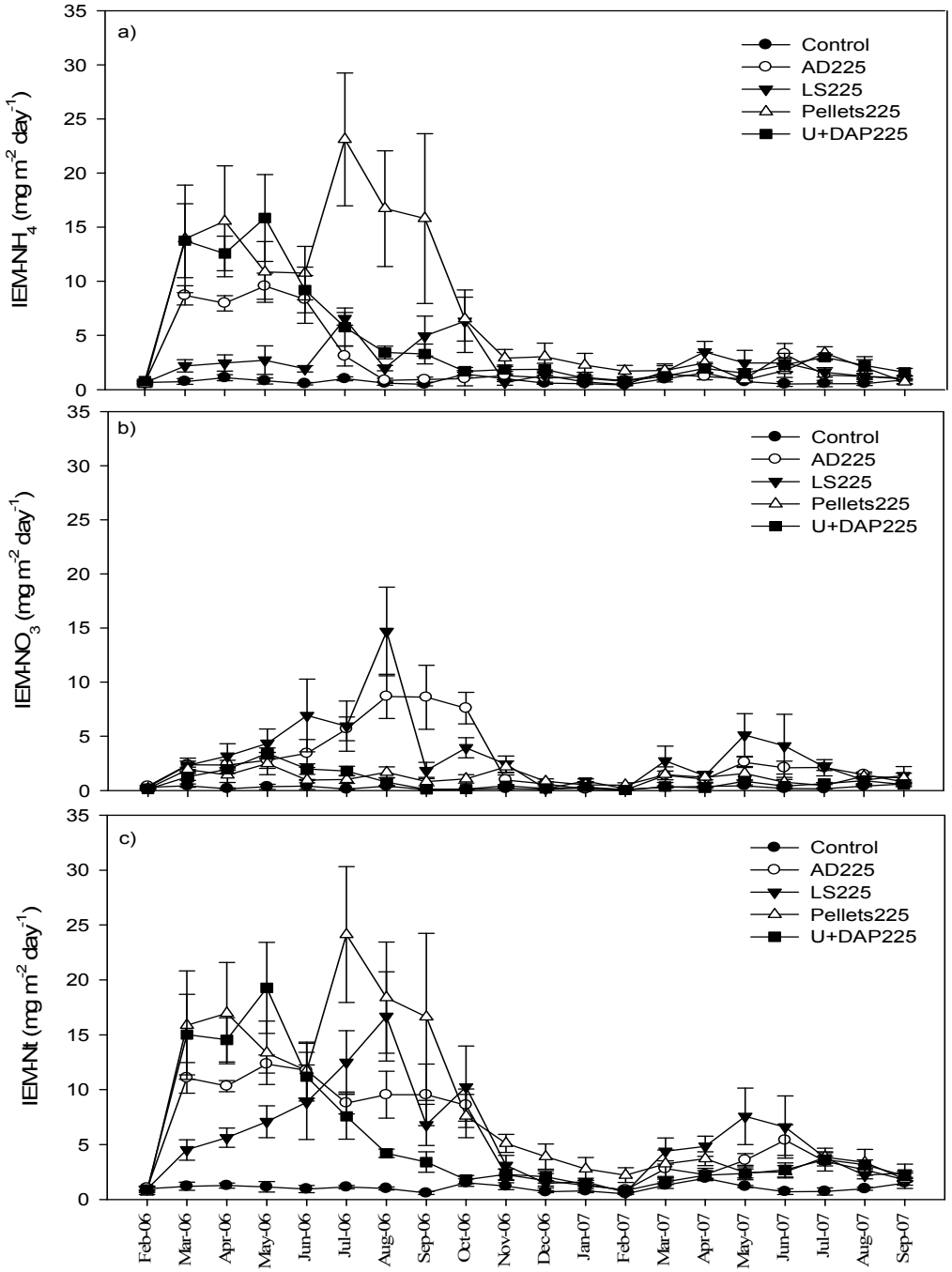


Figure 3.2. Monthly IEM-NH_4(a), IEM-NO_3(b), and IEM-Nt(c) from the forest floor and the upper 10 cm of mineral soil measured for 20-month. Treatments were control, anaerobically digested (AD225), lime stabilized (LS225), pelletized biosolids (Pellet225), and Urea+DAP, applied during March of 2006 at the rate of 225 kg PAN ha^{-1}. Brackets indicate ± SE.

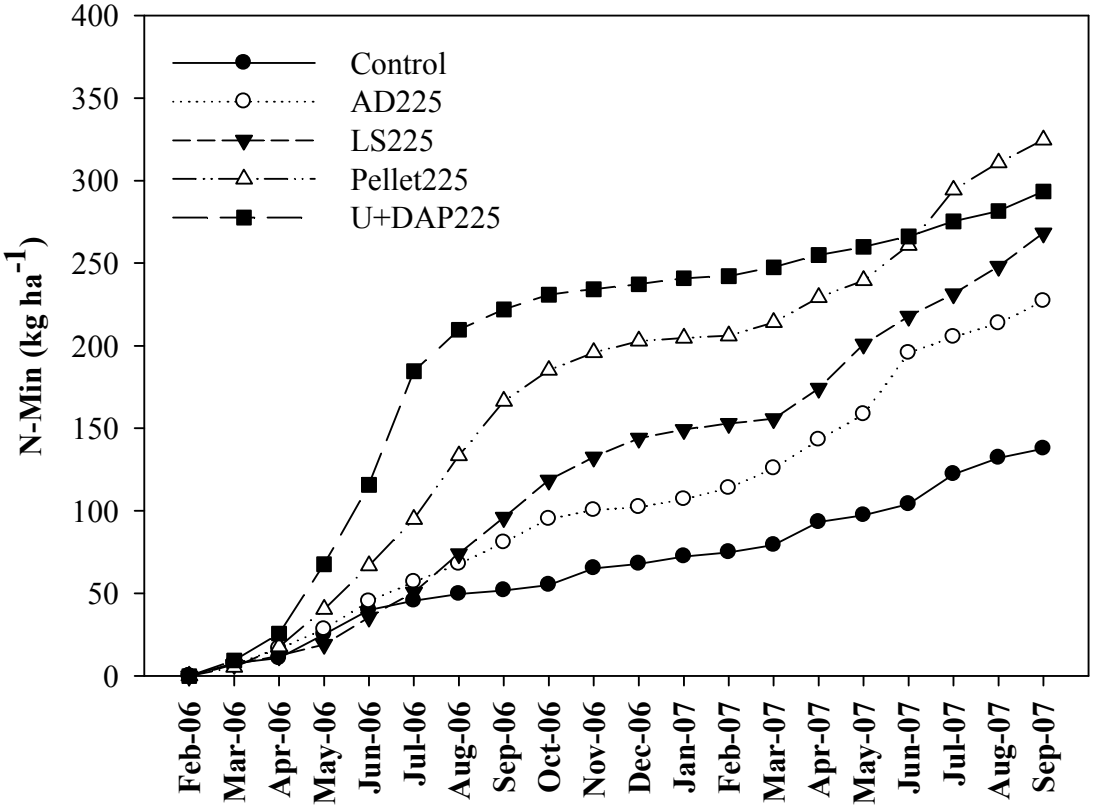


Figure 3.3. Cumulative N mineralization measured in the surface mineral soil over a 20-month sampling period after surface applied biosolids on March 2006 to a 17 year-old loblolly pine plantation in Amelia County, VA. Application rate was 225 kg PAN ha^{-1}. Treatments were control, anaerobically digested (AD225), lime stabilized (LS225), pelletized (Pellet225), and urea + DAP.

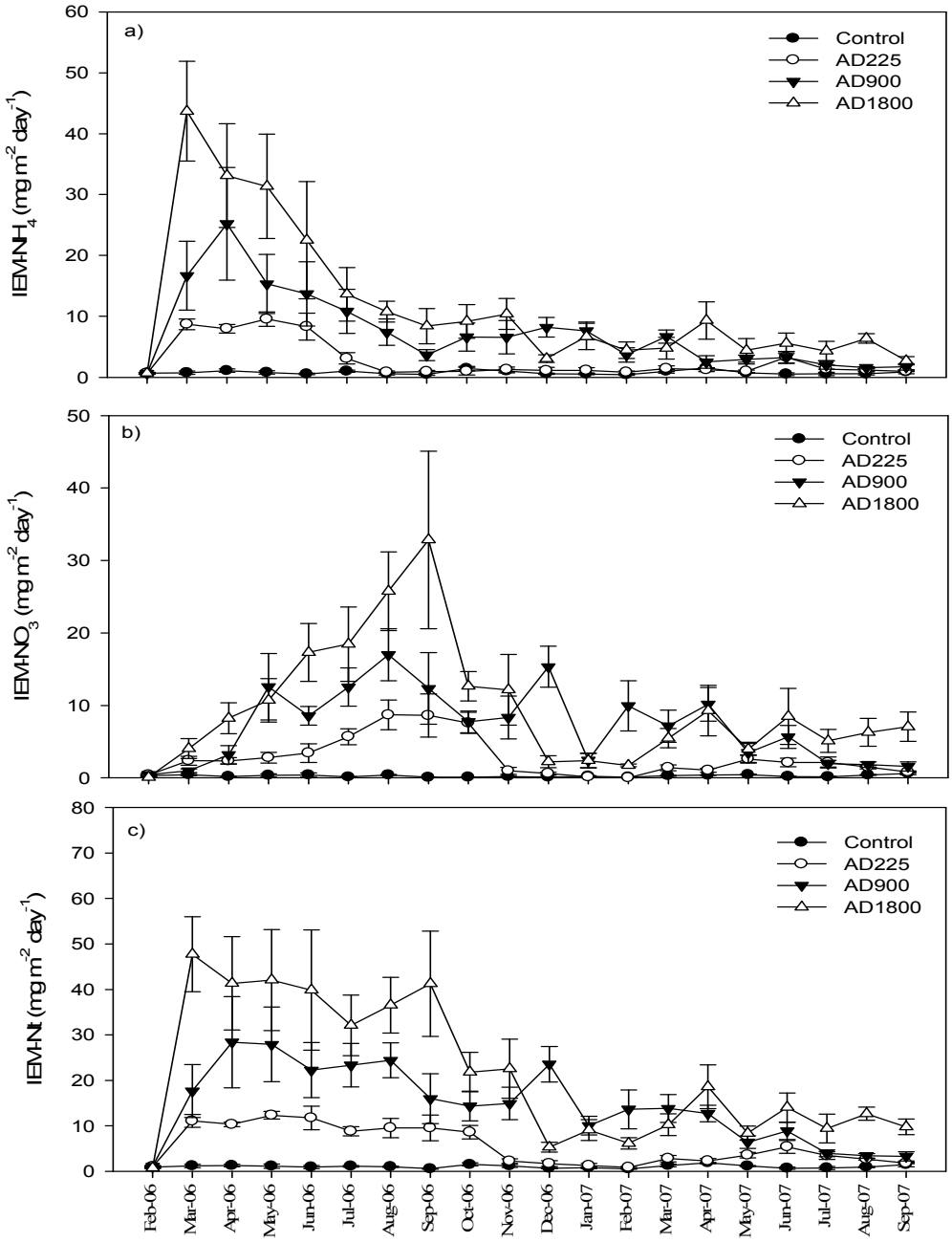


Figure 3.4. Monthly IEM-NH_4(a), IEM-NO_3(b), and IEM-Nt(c) from the forest floor and the upper 10 cm of mineral measured for 20-month. Treatments were control and anaerobically digested biosolids applied during March of 2006 at the rates of 225 (AD225), 900 (AD900), and 1800 (AD1800) kg PAN ha^{-1}. Graph units are in a different scale. Brackets indicate ± SE.

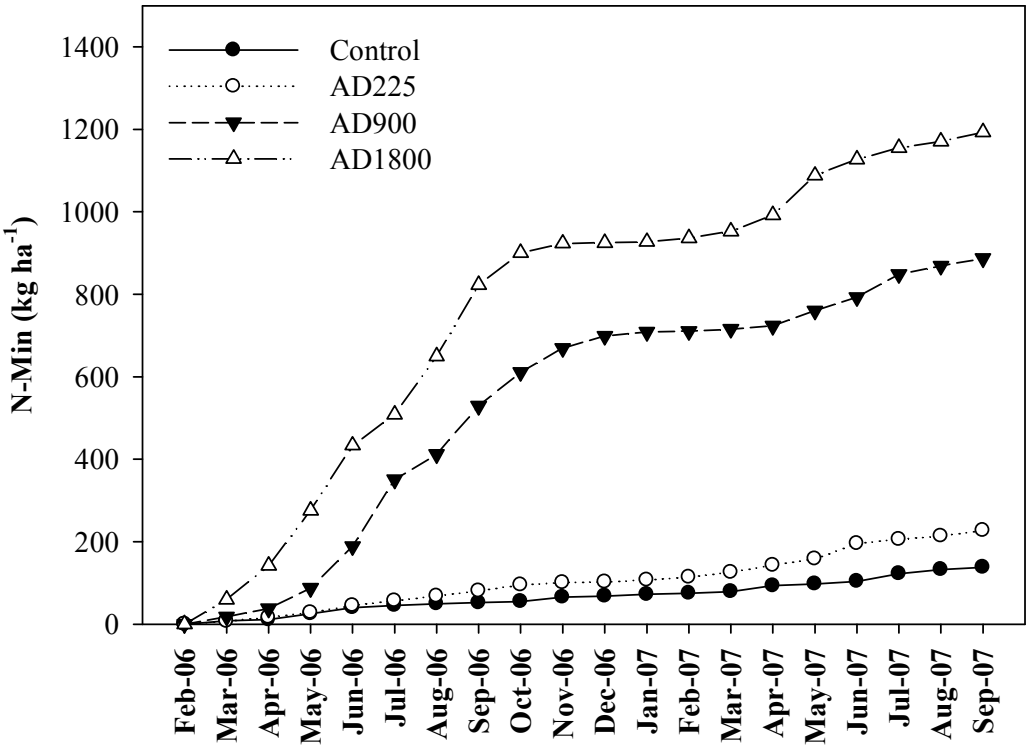


Figure 3.5. Cumulative N mineralization measures in the surface mineral soil over a 20-month sampling period after surface applied anaerobically digested biosolids on March 2006 to a 17 year-old loblolly pine plantation in Amelia County, VA. Application rates were 225 (AD225), 900 (AD900), and 1800 (AD1800) kg PAN ha^{-1} .

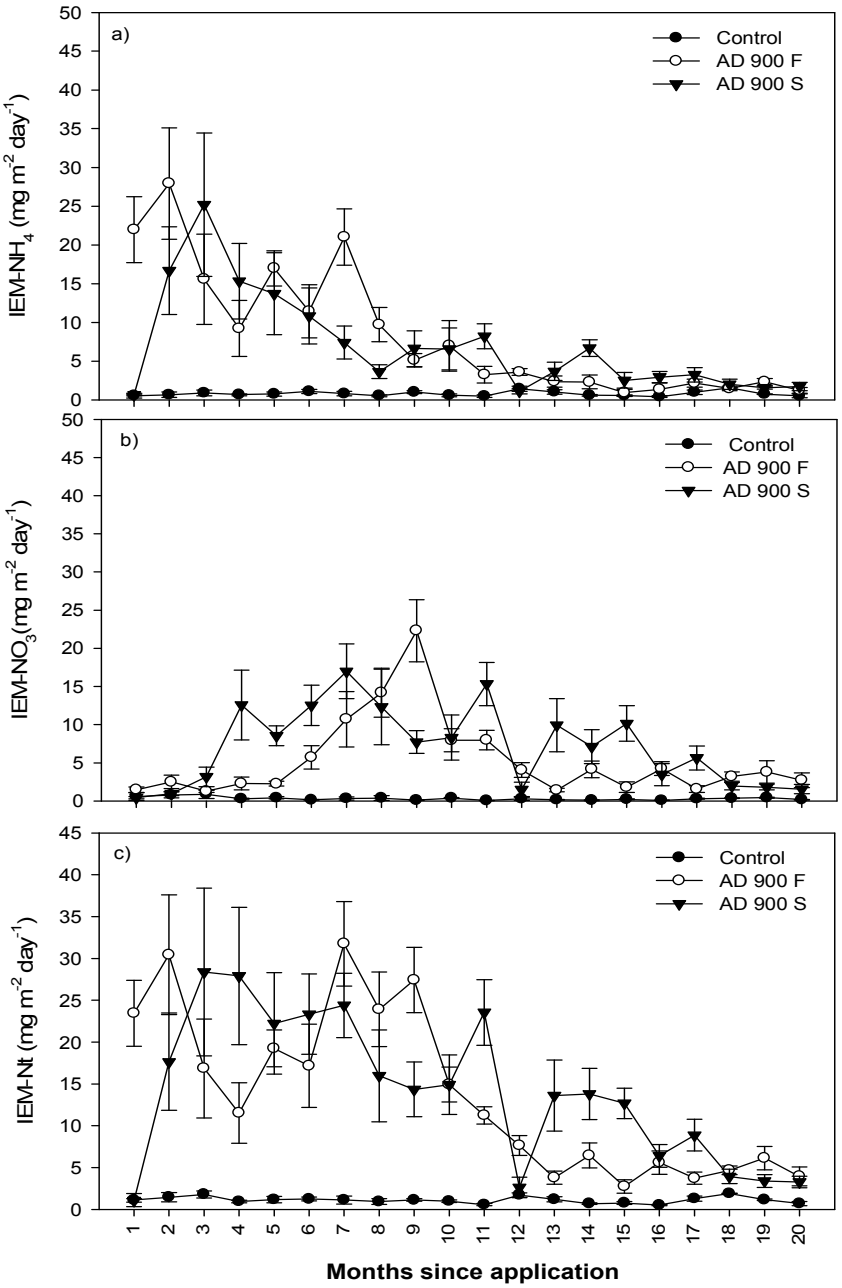


Figure 3.6. Monthly IEM-NH_4 (a), IEM-NO_3(b), and IEM-Nt(c) from the forest floor and the upper 10 cm for a period of 20-month. Treatments are anaerobically digested biosolids applied during November 2005 (fall), and March (spring) of 2006 at a rate of 900 kg PAN ha^{-1}, and reported in units of mg-N m^{-2} day^{-1} of IEM surface. Brackets indicate ± SE.

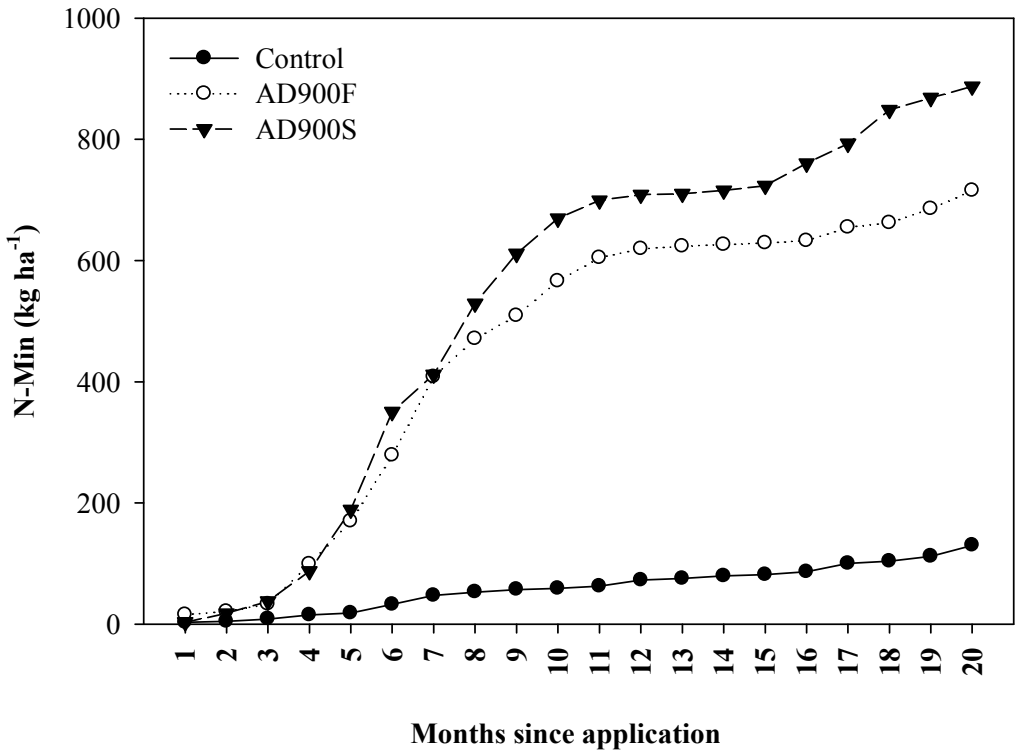


Figure 3.7. Cumulative N mineralization measured in the surface mineral soil over a 20-month sampling period after surface applied biosolids on November 2005 (fall), and March 2006 (spring) to a 17 year-old loblolly pine plantation in Amelia County, VA. Treatments were anaerobically digested biosolids applied at the rates of 900 kg PAN ha^{-1}.

Chapter 4
Nitrogen Leaching Following Application of Biosolids in a Loblolly Pine (*Pinus taeda* L.) Plantation in the Virginia Piedmont.

Abstract

Application of biosolids to pine plantation in the Virginia Piedmont is a feasible alternative to application on cropland and pasture. Biosolids are a source of N to forests that is comparable to traditional inorganic fertilizers such as urea. However, leaching of nitrate-N (NO_3-N) into groundwater following application of biosolids is a major concern in the Chesapeake Bay watershed. The objectives of the study were to determine changes in N availability and leaching through time after one application of (1) different type of biosolids, (2) different rates of biosolids application, and (3) different season of application of biosolids. The study was established in Virginia at a 17-year-old loblolly pine (*pinus taeda*) plantation in Amelia County, VA. Anaerobically digested (AD225), lime stabilized (LS225) and pelletized (Pellet225) biosolids, and a urea plus diammonium phosphate (U+DAP225) were applied at a rate of 225 kg ha^{-1} of Plant Available Nitrogen (PAN) between March 5th and 10th, 2006. Anaerobically digested biosolids were also surface applied at the rates of 900 kg ha^{-1} PAN and 1800 kg ha^{-1} PAN (AD900S, and AD1800) to compare the effects of biosolids application rates. Anaerobically digested biosolids at a rate of 900 kg ha^{-1} PAN was also applied on November 5th, 2005 (AD900F) to compare the effects of season of application of biosolids. Surface soil was sampled once a month to a depth of 15 cm, and soil solution samples were collected at 80 cm depth on a monthly basis.

Land application of different types of biosolids at a rate of 225 kg PAN ha^{-1} significantly increased KCl extractable N in the surface mineral soil and NO_3-N concentration in lysimeter samples collected at 80 cm. Peak NO_3-N concentration in soil solution collected at 80 cm were

of 3.2 mg L^{-1} in the Pellet225, 1.4 mg L^{-1} in the LS225, 1.2 mg L^{-1} in the AD225 and 1.9 mg L^{-1} in the U+DAP225. High application rates of biosolids increased KCl extractable N and N concentration in soil solution in lysimeters. Nitrate-N in lysimeters were frequently above 10 mg L^{-1} with peaks of 97.8 mg L^{-1} and 179.8 mg L^{-1} in the AD900 and AD1800 treatments, respectively. Spring application significantly increased soil extractable N in comparison to the fall application. Nitrate-N in soil solution at 80 cm was significantly higher in the AD900S compared to the AD900F, with peaks of 42.6 mg L^{-1}, and 96.8 mg L^{-1}, respectively. The results from this study indicated that application of biosolids at the rate of 225 kg PAN ha^{-1} increased N availability without increasing the potential for groundwater pollution. Nitrate-N movement through soil profile is likely to occur through preferential flow paths generated from old roots channels and soil cracks.

4.1. Introduction

Nitrogen (N) availability throughout the forest rotation plays a major role in forest nutrition and productivity (Fox et al. 2007a). Nitrogen and phosphorus (P) are limiting nutrients in most southern pine plantations, and fertilization with urea and diammonium phosphate (DAP) is a regular forest management prescription (Albaugh et al. 2007). In loblolly pine, volume growth increases up to 30 % with fertilizer applications of 225 kg of N and 28 kg of P ha^{-1} (Fox et al. 2007a). Application of biosolids to forestland have the potential to improve soil productivity and nutrient availability (Henry et al. 1999). Several studies reported positive changes in forest nutrient availability as a consequence of biosolids application (Brockway 1983; Hallett et al. 1999; Magesan et al. 1998; Wang et al. 2004a).

In the Chesapeake Bay area, N losses from agricultural activities are recognized as a

major pollutant in surface water and coastal marine environments (Boesch et al. 2001). Groundwater pollution through nitrate-N (NO_3-N) leaching is one of the main concerns about land application of biosolids (Evanylo 1999a). For example, accumulation of NO_3-N in water bodies, such as lakes, rivers and estuaries can result in eutrophication (Howarth 1988). The maximum amount concentration for safe drinking water in the USA is limited to 10 mg NO_3-N (USEPA 1986).

Nitrate-N loading in excess of plant demands increases the potential for NO_3-N leaching. NO_3-N leaching also increases soil acidification and losses of cations (Johnson et al., 1991). Studies on forest and agriculture systems indicate that application rates (Aschmann et al. 1992), soil textures (Evanylo 2003), biosolids incorporation time (Gove et al. 2002; Jaynes et al. 2003), and the presence of understory vegetation (Wang et al. 2004b), directly affect NO_3-N mobility through soil profile. In order to safely apply biosolids to forestland and minimize the potential for nitrate leaching, plant N availability can be estimated based on the initial N concentration in biosolids, the application method, type of biosolids, and the plant N demand (Evanylo 1999b; Henry et al. 1999; USEPA 1995).

Nitrogen in biosolids is primarily organic-N and ammonium (NH_4-N), with very low concentration of NO_3-N (Gilmour et al. 2003; Sommers 1977). Most of the N added with biosolids is organic N that needs to be mineralized in order to be available for plant uptake. Biosolids release N at different rates depending factors that affect mineralization rates including their composition, application method, and soil conditions. The wastewater treatment processes include aerobic and anaerobic stabilization, heat-dried, composted, and addition of chemical that affect nutrient release after land application (Wang et al. 2008). Biosolids treatments, like lime addition, affected soil pH, reducing soil microbial activity and mineralization (Simmons et al.

1996). For example, anaerobically digested biosolids had lower N mineralization rates than aerobically digested biosolids as a consequence of the stabilization processes (Garau et al. 1986).

Surface application of biosolids is the most common application method used in forests. The initial N added as NH_4-N with the biosolids initially increase soil N availability, although a portion may be lost through volatilization, denitrification or immobilization (Henry et al. 1999; Robinson and Polglase 2000; Sommers et al. 1979). Longer term increases in N availability depend on mineralization (Kelty et al. 2004; Robinson et al. 2002).

Specific forest soil characteristics like acidity, mineralogy and carbon content directly affect N mobility (Eick et al. 1999; Gilmour 1984; Johnson and Cole 1980; Silva et al. 2005). Nitrate-N produced after biosolids application could be lost from negatively charged soils (Gilmour et al. 2003; Rowell et al. 2001). In contrast, variable charged soils have the potential adsorb significant quantities of NO_3-N (Strahm and Harrison 2007). Although NO_3-N leaching have seldom been observed in southern forest soils (Binkley et al. 1999) it is possible to occur in forest soils that contain preferential flow paths such as decaying root channels (Franklin et al. 2007).

There are no field experiments on loblolly pine plantations located in the Piedmont, that compare the effect of different type of biosolids, and how increasing rates affect N availability and leaching potential over time. In the previous chapter we reported significant effects of biosolids application on N availability, especially after high application rates. The objective of this study was to measure and compare the effect of different type of biosolids on N availability and leaching in a 17-year-old loblolly pine plantation. Nitrate sorption capacity in this soil was measured to determine the potential of NO_3-N to leach through these soils following land application of biosolids. It is necessary to consider the movement and N losses through leaching.

The specific objectives of the study were to determine changes in surface NH_4-N and NO_3-N availability, and leaching through time after one application of different (1) type of biosolids, (2) three different application rates of the same type of biosolids, and (3) different season of biosolids application.

4.2. Material and Methods

4.2.1. Study Area

The study was established in the summer of 2005 in Amelia County northeast of Blackstone, VA (37° 13' N, 77° 48' W). The site is located in the Piedmont physiographic province and supported a 17-year-old loblolly pine plantation. The stand was thinned in 2005 using a combination of fifth-row removal and low thinning between the removed rows. The mean annual temperature is 14 °C, with a mean of 4 °C in January and 25 °C in July. The mean annual precipitation is 113 cm with July and August being the wettest. The mean snowfall is 30 cm concentrated between December and March (15-year period). The local climatologic recent records were obtained from the closest weather station in Blackstone, VA which was approximately 40 km from the study site. During the month of the spring application (March 2006), precipitation was only 0.68 cm, which was below the historical average 8.1 cm for the area (Fig 4.1). Conditions were near normal throughout most of the spring and summer of the same year, while rainfall from August to November 2006 was greater than normal.

The soil at the site is the Appling series (Fine, kaolinitic, thermic Typic Kanhapludults). Slopes range from 2% to 5%. The thin Ap layer shows evidence of surface soil erosion caused by past agriculture practice, and in some areas is mixed with the argillic Bt horizon. Soil samples collected at four different depths, from 0 to 20 cm, 20 to 40 cm, 40 to 60 cm, 60 to 80 cm prior to

treatments were analyzed for total C and N by combustion using a CNS analyzer (Elementar America, Inc, Laurel, NJ), and Mehlich-1 extractable P, K, Ca, Mg using a ICAP-AES following the Virginia Cooperative Extension Methodology (Donohue and Heckendorn 1994; Mehlich 1953). Soil pH was determined in a 1:1 soil:water ratio. Soil cores were collected with a bulk density hammer and subsequently oven dried and weigh to measure bulk density (Table 4.1).

4.2.2. Experimental Design and Treatments

The experimental design was a randomized complete block design with four blocks and eight treatments comparing biosolids type, granular fertilizer, rates, and season of application. Thirty-six plots of 0.45 ha (150 x 30 m) separated by a 30 m of untreated buffer area were established in September 2005. Three different types of biosolids were evaluated; lime stabilized, anaerobically digested, and pelletized biosolids. The anaerobically digested material was obtained from the Alexandria, VA and Back River, MD wastewater treatment facilities, for the fall and spring application, respectively. The lime stabilized biosolids were obtained from the Blue Plains wastewater treatment facility in Washington, DC. The pelletized biosolids were obtained from the Synagro pelletized facility in Back River, MD. The granular fertilizer treatments were a mix of conventional urea-N (46-0-0) and DAP (18-46-0) obtained from Southern States Cooperative, Inc., (Christiansburg, VA). Physical and chemical properties of the biosolids are presented in Table 4.2.

Anaerobically digested, lime stabilized, pelletized biosolids and a conventional inorganic fertilizer were applied at a rate of 225 kg ha^{-1} Plant Available Nitrogen (PAN). In order to understand the consequences of application rates greater than the recommended rate of 225 kg PAN ha^{-1}, anaerobically digested biosolids were added at 900 and 1800 kg PAN ha^{-1}. These

treatments were applied between March 5th and 10th, 2006. To test the impact of season of application timing (fall vs. spring), the anaerobically digested biosolids were also applied at 900 kg PAN ha^{-1} on November 5th, 2005. Treatments descriptions and final application rates are listed in Table 5.3.

Biosolids application rates were estimated based on the average N concentration of samples analyzed during the six previous months before the beginning of the study, and corroborated with field samples. The treatments rates were based on PAN approach, determined according to recommendations established for VA when biosolids are surface applied (Evanylo 1999b).

$$\mathbf{PAN = NO_3\text{-}N + \mathit{Kvol}\ (NH_4\text{-}N) + \mathit{Kmin}\ (Org\text{-}N)}$$

Where:

PAN = Kg of Plant Available N dry^{-1} Mg biosolids

NO_3-N = Kg nitrate-N dry^{-1} Mg biosolids

Kvol = volatilization factor, or plant-available fraction of NH_4-N (lime stabilized = *0.25*, anaerobically digested = *0.5*)

NH_4-N =Kg ammonium-N dry^{-1} Mg biosolids

Kmin = mineralization factor, or plant-available fraction of Org-N (lime stabilized = *0.3*, and anaerobically digested = *0.2*)

Org-N = Kg organic-N dry^{-1} Mg biosolids (estimated by organic N = total Kjeldahl-N - NH_4-N)

The biosolids were transported from each wastewater treatment plant, piled at the site and applied during the same day. Biosolids were surface applied using a skidder with a side

discharge spreader that went through the previous thinned corridors in the stand. The biosolids were not tilled into the soil. Four plastic collection trays (60 x 20 cm) were installed in each plot prior to application. The biosolids collected in each treatment were weighed, and moisture content was determined after the application. Samples were collected from the trays in the field and transported to A&L Eastern Agricultural Laboratories (Richmond, VA) for routine biosolids test analysis for total and volatile solids (SM2540G), nitrate-N (SM4500- NO_3-F) (APHA 1998), pH according to SW 846-9045C (USEPA 2002), and calcium carbonate equivalent (CCE) using AOAC 955.01 (Kane 2000). Total Kjeldahl nitrogen (USEPA 351.3) and ammonium-N (USEPA 350.2) (USEPA 1983). Phosphorus (P), potassium (K), sulfur (S), calcium (Ca), magnesium (Mg), sodium (Na), iron (Fe), manganese (Mn), cadmium (Cd), copper (Cu), lead (Pb), molybdenum (Mo), nickel (Ni), and zinc (Zn) were measured according to SW846-6010B (USEPA 2002) (Table 4.2).

Conventional granular fertilizer treatments were broadcast at the same time using a backpack spreader so the fertilizer evenly covered the forest floor. The competing herbaceous understory vegetation was removed chemically using a foliar application of 5% Round-up Pro ™ (Monsanto Co, St Louis, MO) applied with a backpack sprayer in all plots during the summer of 2005 and 2006.

4.2.3. Soil Extractable Nitrogen

Soil KCl extractable NH_4-N (KCl-NH_4) and NO_3-N (KCl-NO_3) were measured in soil samples collected using cores made of polyvinyl chloride pipe (3.8 diameter by 20.3 cm in length). In each treatment plot, a soil core were randomly located and inserted, vertically into the mineral soil to a depth of 15 cm. The core was immediately removed and stored in a cooler and

transported to the laboratory in Blacksburg, VA. The soil was removed from the core and sieved through a #10 mesh screen. The sieved soil was stored at 4 °C in plastic bags until extraction. At the time of soil extractions, 5 g. of field moist soil was dried at 105°C for 24 hours, and reweighed to determine moisture corrections. Another 5 g. sample of soil from the same sample was placed in a centrifuge tube with 50 ml of 2 M KCl and shaken on a reciprocating shaker for 1 hour. Samples were centrifuged for 10 minutes, filtered through Whatman #42 paper, and the extracts were transferred to scintillation vials and frozen until analyzed for NH_4-N and NO_3-N. The KCl extracts were analyzed colorometrically for NH_4-N (USEPA Method 350.1) and NO_3-N (USEPA Method 353.2) using a TRAACS 2000 Auto Analyzer (SEAL Analytical, Mequon, WI). Soil extractable N was converted to a kg per ha based on surface soil bulk density and cores depth.

4.2.4. Soil Solution Analysis

Soil solution samples were collected with porous cup tension lysimeters (Soil moisture Equipment Corporation, Santa Barbara, CA). These types of lysimeters have been used on previous leaching studies with biosolids (Brockway and Urie 1983; Medalie et al. 1994). The porous ceramic cups were attached to a 1 m long polyvinyl chloride (PVC) pipe. Two lysimeters were installed in each of the thirty-six plots at a depth of 80 cm, which was generally in the BC or C horizon immediately below the Bt horizon, All lysimeters were installed during the summer of 2005, five weeks before fall biosolids application. Samples were collected on a monthly basis. Lysimeters were evacuated to a tension of -50 kPa and the soil solution collected after 24 hours. The soil solution was placed in a cooler and then frozen until analysis. Samples were analyzed for NH_4-N (L-NH_4) and NO_3-N (L-NO_3) by colorimetry using a TRAACS 2000 Auto Analyzer

(SEAL Analytical, Mequon, WI).

4.2.5. Nitrate Sorption Capacity

Previous studies with similar acidic soils have shown that anion adsorption capacity affects NO_3-N movement (Eick et al. 1999; Strahm and Harrison 2008). In order to understand how soil properties could affect NO_3-N leaching, we collected soil samples from the unfertilized control plot at the depths of 0-20 cm, 20-40 cm, 40-60 cm, and 60-80 cm and determined the NO_3-N sorption capacity using a batch equilibration technique (Kinjo and Pratt 1971; Kowalenko and Yu 1996; Strahm and Harrison 2006). Soil was air-dried and sieved before laboratory procedures. Five-gram subsamples of each soil were equilibrated with 20 ml of six increasing concentrations of NH_4NO_3 (6, 12, 34, 48, 96 and192 mg NO_3-N L^{-1}). The soil sample and NO_3-N solution mixture was shaken for 1 hour at room temperature. Following equilibration, the mixtures were centrifuged and the supernatant was filtered through Whatman no. 42 filter paper. Concentrations of NO_3-N in the solution were determined using a TRAACS 2000 Auto Analyzer (SEAL Analytical, Mequon, WI). Soil and solution equilibrations were performed for each soil depth, and replicated 4 times. For each soil depth, linear regression of the equilibration-solution against the NO_3-N treatment concentration was used to determine the proportion of NO_3-N recovered, using the Modified Hyperbola I nonlinear regression from SigmaPlot ®10.0 (Systat software inc, Germany). The maximum level of NO_3-N sorbed (N_{max}), and the affinity of NO_3-N with the mineral surface (*b*) were determined using the Langmuir isotherm equation:

$$\frac{\text{mmol sorbed}}{\text{Kg soil}} = \frac{b\text{N}max\ (\text{mmol in final solution})}{1 + b\ (\text{mmol in final solution})}$$

4.2.6. Statistical Analysis

All statistical analyses were conducted using SAS statistical software (SAS Institute, Cary, NC). Analysis of variances (ANOVA) using the mixed models procedure with repeated measures were performed to test the effect of treatments, time, and the interactions on monthly KCl-NH_4, KCl-NO_3, L-NH_4, and L-NO_3. Variance-covariance structures were examined to determine the best model for the repeated measures (Littell et al. 2006). Tests for normality, linearity, and constant variance of the residuals were performed. Logarithmic transformations were necessary to ensure the validity of the assumptions. Results are presented in untransformed values. For significant treatment by time interaction ($P<0.05$) in the ANOVA analysis, treatments were compared using Fisher-LSD test at $\alpha = 0.05$.

To permit a direct comparison of the two seasons of application through time on KCl-NH_4, KCl-NO_3, L-NH_4, and L-NO_3 the data were normalized using the number of months since biosolids application rather than the actual sampling date. Selected contrast comparisons were performed to compare the main effect of treatments on N availability and leaching.

4.3. Results

Application of the three different types of biosolids applied in March 2006 at the 225 kg PAN ha^{-1} application rate had a significant effect on N availability in the surface mineral soil. There was a significant treatment by time interaction effect on KCl-NH_4, KCl-NO_3, L-NH_4, and L-NO_3 (Table 4.4).

4.3.1. Effect of Biosolids Type

4.3.1.1. Soil Extractable KCl-NH_4 and KCl-NO_3

Soil KCl-NH_4 and KCl-NO_3 were significantly higher in the biosolids treatments and the conventional fertilizer treatments than the control plots (Table 4.5). Soil KCl-NH_4 was highest in the Pellet225 compared to all the other treatments. No significant differences were detected between AD225 and the LS225 treatments (Table 4.5). In contrast, soil KCl-NO_3 in the Pellet225 was significantly lower than the AD225. No significant differences were detected among the other treatments (Table 4.6).

Soil KCl-NH_4 responses at each sampling date showed that the Pellet225, AD225, and the U+DAP225 treatments increased immediately after treatments application (Table 4.6, Fig 4.2.a). The peak response in soil KCl-NH_4 in the Pellet225 was 87.8 kg ha^{-1} in July 2006, 60.9 kg ha^{-1} in the U+DAP225 in May 2006, 21.5 kg ha^{-1} in the LS225 in July 2006, and 24.7 kg ha^{-1} in the AD225 treatment in May 2006.

From March to May 2006, soil KCl-NH_4 was similar between the Pellet225, and the U+DAP225 treatments. However, soil KCl-NH_4 declined in the U+DAP225 after June 2006, but remained elevated in the Pellet225 until September 2006 (Table 4.6, Fig 4.2.a). Soil KCl-NH_4 in the AD225 was significantly greater than the control in March and May 2006, and then remained close to control levels (Table 4.6, Fig 4.2.a). The AD225 and the Pellet225 were significantly greater than the control from December 2006 until January (AD225) or February (Pellet225) 2007. The Pellet225 was also greater than the control in April, July and August 2007.

Soil KCl-NH_4 in the LS225 followed a different pattern than in the other biosolids treatments. Initially soil KCl-NH_4 was not different than the control. However it increased in July 2006 and was significantly greater in September 2006, and then late in December 2006, and August 2007 (Table 4.6, Fig 4.2.a).

The application of biosolids also significantly affected KCl-NO_3, however the response occurred more slowly than it did for KCl-NH_4 and not until June 2006, all the treatments were significantly greater than the control (Table 4.7, Fig 4.2.b). Peak soil KCl-NO_3 in the AD225 treatment was 19.2 kg ha^{-1} in September 2006, 24 kg ha^{-1} in the LS225 in August 2006, 15.1 kg ha^{-1} in the Pellet225 in the October 2006, and 29.3 kg ha^{-1} in the U+DAP225 in July 2006. Biosolids KCl-NO_3 followed a similar pattern that differed from the U+DAP225 (Fig 4.2.b). Soil KCl-NO_3 was significantly higher than the control from April (AD225, Pellet225), or June (LS225) until December 2006. Soil KCl-NO_3 in biosolids treatments was similar and significantly greater than the control periodically during the same months during 2007. The U+DAP225 treatment started to decline in September 2006, and remained close to control levels until March 2007, with the exception of December 2006. Soil KCl-NO_3 in the U+DAP225 was significantly greater than the control during the months of April, June, July and September of 2007 (Fig 4.2.b).

4.3.1.2. NH_4-N and NO_3-N leaching

No samples were collected in the lysimeters from any treatments between May and July 2006, and June and August 2007 because the soil was too dry to extract soil solution.

Biosolids type had no effect on NH_4-N concentration in soil solution sampled from lysimeters (Table 4.5), and soil solution N concentrations were no different from the control treatment at any sampling date (Table 4.8). Ammonium-N concentrations in soil solution were between 0.1 and 0.57 mg L^{-1}, with several samples below detection limits (Table 4.8, Fig 4.3.a).

There was a significant treatment effect on L-NO_3, although mean values were below 1.1 mg L^{-1} in all the treatments. The average L-NO_3 in the LS225 and the Pellet225 treatments were

significantly higher than the control. Nitrate-N concentration in soil solutions sampled in the Pellet225 was significantly higher than the AD225 and the U+DAP225 (Table 4.5).

Initial L-NO_3 in all the treatments remained low until August 2006. The Pellet225 was significantly higher than the control from September 2006 to December 2006, and then in March and April 2007. The peak L-NO_3 sampled from lysimeters was 3.2 mg L^{-1} in the Pellet225 in November 2006, 1.4 mg L^{-1} in LS225 in August 2006, 1.2 mg L^{-1} in October 2006 in the AD225, and 1.9 mg L^{-1} in U+DAP225 treatment during October 2006 (Fig 4.3.b).All the other biosolids treatments remained close to control levels throughout most of the study, with the exception of the soil solution sampled in September 2006 in the LS225 treatment (Table 4.9, Fig 4.3.b).

4.3.2. Effect of Biosolids Application Rates

4.3.2.1. Soil KCl-NH_4 and KCl-NO_3

Increasing biosolids rates significantly increased soil KCl-NH_4 and KCl-NO_3. Mean values for the 21-month study period were significantly greater in the AD1800, and AD900 treatments relative to the AD225. In contrast, there was no difference between the AD1800 and the AD900 (Table 4.5).

The application of higher rates of biosolids significantly increased soil KCl-NH_4 during most of the sample dates (Table 4.10). The peak soil KCl-NH_4 content was 108.8 kg ha^{-1}, 60.2 kg ha^{-1}, and 24.7 kg ha^{-1} in the AD1800, AD900, and AD225, respectively (Table 4.10). Soil KCl-NH_4 declined through time at all three rates; however it remains elevated longer in the AD1800, and AD900 treatments. Soil KCl-NH_4 concentration in the AD900, and the AD1800 treatments remained generally higher than the AD225 until January 2007. The KCl-NH_4 in the AD900

treatment was significantly higher than the AD225 again between May and July 2007. The AD1800 was higher than the AD225 treatment in July 2007 (Table 4.10, Fig 4.5.a).

Soil KCl-NO_3 content increased more gradually than the KCl-NH_4 (Fig 4.5.b). Soil KCl-NO_3 content peaked at 89 kg ha^{-1}, 62.7 kg ha^{-1}, and 19.2 kg ha^{-1} in September 2006 in the AD1800, AD900, and AD225 treatments, respectively. Soil KCl-NO_3 in the AD900 and AD1800 treatments remained higher than the AD225 from June 2006 to March 2007 (AD900) or April 2007 (AD1800) (Table 4.11, Fig 4.5.b). The AD1800 and AD900 were higher than the AD225 occasionally during the second year (Fig 4.5.b). The two highest rates had similar patterns and were not significantly different, with the exception of April and October 2007 (Table 4.11).

4.3.2.2. NH_4-N and NO_3-N Leaching

Concentration of NH_4-N and NO_3-N in soil solution measured in lysimeters in the AD1800 and AD900 were significantly different than AD225 treatment. There was no difference between the AD1800 and the AD900 treatments (Table 4.5). The AD1800, AD900, and AD225 treatments had a mean L-NH_4 in soil solutions below 0.8 mg L^{-1} throughout the 21-month period (Table 24, Figure 7a). The AD1800 had a peak of 2.1 mg L^{-1} in September 2006, and the AD900 peak was 2 mg L^{-1} in November 2006 (Table 4.12). The AD1800 and AD900 treatments had significantly greater L-NH_4 than the AD225 from August 2006 to November (AD900) or December 2006 (AD1800) (Table 4.12, Fig 4.6.a).

Mean L-NO_3 was 37.4 mg L^{-1} in the AD900 treatment, and 70 mg L^{-1} in the AD1800 treatment throughout the whole sampling period (Table 4.13). Concentration of NO_3-N in solution was much higher in the AD1800 and the AD900 plots compared to the AD225 treatment in most of the sampling time (Table 4.13, Fig 4.5.b). The AD1800 reached 179.8 mg L^{-1} in

September 2006 and the AD900 peaked 97.9 mg L^{-1} in November 2006. Both treatments L-NO_3 in lysimeters declined after May 2007 (Fig 4.6.b).

4.3.3. Effect of Season of Biosolids Application

4.3.3.1 Soil KCl-NH_4 and KCl-NO_3

Season of application of anaerobically digested biosolids at 900 kg PAN ha^{-1} significantly affected soil KCl-NH_4 and KCl-NO_3. Average soil KCl-NH_4 and KCl-NO_3 was significantly greater in the AD900S than the AD900F (Table 4.5). Soil KCl-NH_4 was similar during the first year after application, with the exception of the month 5, 7, and 9. The AD900S treatment significantly increased soil KCl-NH_4 between the month 15 and 19 (Table 4.14). The peaked in the AD900F was earlier than the AD900S. The AD900F reached 77.2 kg ha^{-1} during month 2, and the AD900S reached 63.5 kg ha^{-1} during month 7 (Table 4.14, Fig 4.7.a).

Soil KCl-NO_3 was similar in the AD900F and AD900S treatments through most of the sampling date. The AD900S was significantly higher than the AD900F during the months 5, 7, and 8 following treatments application (Table 4.15). The peak in the AD900S was 62.7 kg ha^{-1} during month 8, and 47.8 kg ha^{-1} in the AD900F during month 12.

4.3.3.2. NH_4-N and NO_3-N Leaching

Due to the dry soil, no samples were collected from the lysimeters between month 7 and 9, and month 20 and 21 in the AD900F treatments, and between month 4 and 6, and 17 to 19 in the AD900S.

The mean L-NH_4 concentration measured in lysimeters was significantly greater in the AD900F than in the AD900S (Table 4.5). The AD900F had a peak value of 39.2 mg L^{-1} two

months after application. The AD900S had a L-NH_4 peak of 2.9 mg L^{-1} in month 3 (Table 4.16, Fig 4.8). In contrast, the average L-NO_3 was significantly higher in the AD900S treatment (Table 4.5). Peak L-NO_3 concentration in soil solution was 42.6 mg L^{-1} in month 10 in the AD900F, and 97.9 mg L^{-1} in the AD900S in month 13 (Table 4.17). The mean NO_3-N concentration in soil solution collected from lysimeters was 6.1 mg L^{-1} in the AD900F, and 37.5 mg L^{-1} in the AD900S throughout the whole study time (Table 4.17, Fig 4.8).

4.3.4. Soil Nitrate Sorption Capacity

Soil at the study site adsorbed substantial amounts of NO_3-N. Nitrate-N sorption capacity was higher in the Bt_1 and Bt_2 layers than in the Ap and BC layers (Table 4.18, Fig 4.9). The total NO_3-N sorption capacity in this soil through 80 cm depth was 7159 kg $ha^{-1.}$ Nitrate-N sorbed to the mineral surface increased with a greater amount of NO_3-N in solution (Fig 4.9). The fraction sorbed was lowest at higher solution concentration. NO_3-N sorption increased in the Bt_1 and Bt_2 horizons in comparison to the Ap and BC horizons as expressed in the maximum sorption capacity in mmol kg soil $^{-1}$, and kg of NO_3-N ha^{-1} based on bulk density measurements from each layer (Table 4.1, Table 4.18, Fig 4.9).

4.4. Discussion

4.4.1. Effect of the Biosolids Type on N Availability and Leaching

We found that application of biosolids in a loblolly pine plantation increased surface soil N availability (Table 4.6, Table 4.7). Our findings are consistent with previous field studies that reported increase in N availability for red pine (*Pinus resinosa* Ait) and white pine (*Pinus*

strobus L) (Brockway 1983), ponderosa pine (*Pinus ponderosa*) (Powers 1980), western red cedar (*Thuja plicata* D) (Prescott and Zabek 1997), mixed northern hardwoods forest (Hallett et al. 1999) and monterey pine (*Pinus radiata* D Don) (Egiarte et al. 2005; Wang et al. 2004a). Increase in soil N have been observed in loblolly pine plantation following application of different type of biosolids (Corey et al. 1986).

The high average extractable NH_4-N in the Pellet225 and the variability in monthly measurements indicated differences among biosolids type. Surface soil extractable NH_4-N showed an immediate effect in the AD225 and Pellet225 treatments. Similar NH_4-N releases have been observed in other biosolids studies, and are explained by the initial NH_4-N content, and the rapid mineralization of the labile organic N in biosolids (Boyle and Paul 1989; Hallett et al. 1999; Terry et al. 1979; Wang et al. 2003). In the AD225, the first pulse of soil extractable NH_4-N was produced by the initial inorganic N content in the applied biosolids (Table 3.2). In contrast, the source of soil extractable NH_4-N in the Pellet225 came from the mineralization of the organic-N. Rapid N mineralization in pelletized biosolids have been observed on field incubations. Kelty et al. (2004) reported that 26 % of the organic N applied with pelletized biosolids was rapidly mineralized after surface application in red pine forest within the first growing season. Eldridge et al (2008) observed a rapid N release within 2 months after surface application of pelletized biosolids in a field incubation study.

Even though the biosolids contained no NO_3-N initially, soil extractable NO_3-N slowly increased in the surface soil after biosolids application (Fig 4.2.b). This lag period has been observed in other biosolids studies (Aschmann et al. 1992; Beauchamp et al. 1979). The organic N is first mineralized to NH_4-N which can be oxidized to NO_3-N through nitrification (Jussy et al. 2004; Simmons et al. 1996). Soil NO_3-N content peaked late in the summer when tree N

uptake decreased and conditions for nitrifiers are more appropriated (Aschmann et al. 1992). In this experiment, the response in surface soil NO_3-N explained by nitrification, could be affected by the ability of clay mineral to fix NH_4-N (Feigenbaum et al. 1994), the effect of alkaline biosolids on nitrification (Gilmour 1984; Terry et al. 1981), the presence of nitrifiers in biosolids (Burton et al. 1990), the negative impact on nitrification of forest floor acidic conditions (Burton et al. 1990), and the response to inorganic N and herbicides control on nitrification (Gurlevik et al. 2004).

Although the available NO_3-N in the surface soil increased following application of biosolids at the 225 kg ha^{-1} PAN rate, the low concentration in soil solution indicated little NO_3-N movement below the Bt horizon. The average monthly NO_3-N concentrations in lysimeters was under 1.3 mg L^{-1} for all the biosolids treatments at the 225 kg ha^{-1} PAN application rate, with several months below detectable levels. Our results suggested that biosolids could be used as a source of N with low risk for groundwater pollution when biosolids are surface applied at the permitted rate.

Our results agree with several studies that reported low levels of NO_3-N leaching when similar rates of biosolids were surface applied to forests (Kelty et al. 2004; Medalie et al. 1994; Robinson et al. 2002; Wang et al. 2004a). In contrast, high NO_3-N concentration in leachate have been observed when biosolids were added into a trench (Kelty et al. 2004; Medalie et al. 1994), or in a sandy or coarse soil site (Brockway and Urie 1983), or applied as liquid biosolids (Aschmann et al. 1992; Sidle and Kardos 1979). Riekerk (1981) concluded that the presence of forest floor, understory vegetation, type of biosolids, type of soils, and method of incorporation significantly affected the potential for NO_3-N leaching after application of biosolids to forest.

Soil available N from biosolids can also be lost through volatilization, denitrification, uptake by trees and understory vegetation, storage in soil (Henry and Cole 1997). Surface application of biosolids increased N losses through volatilization (Quemada et al. 1998; Terry et al. 1981), decreasing soil N availability and the potential for subsurface NO_3-N leaching (Robinson et al. 2002). Anaerobically digested biosolids were shown to lose 60% of NH_4-N during a five-day period (Beauchamp et al. 1978), and between 71 to 81% within 3 weeks when biosolids were surface applied (Robinson et al. 2002). The total precipitations during the treatments application in March 2006, was particularly dry and conditions were adequate for N volatilization for biosolids.

Trees and understory N uptake decreased N mobility and leaching (Wang et al. 2004a). Previous studies showed increase in understory vegetation in forest following biosolids (Aschmann et al. 1990; Brockway 1983) or conventional fertilizer application (Flint et al. 2008). Pine plantation thinning combined with conventional fertilization increased tree growth (Fox et al. 2007b). In contrast, vegetation removal increased soil N mobility. High levels of NO_3-N leaching have been reported when biosolids were applied to recent cleared forest (Henry et al. 1994; Wells et al. 1985). Robinson et al. (2002) reported little effect of understory vegetation removal on N losses through leaching when anaerobic or aerobic biosolids were surface applied to forest. In this study, they found that most of the N losses were through volatilization.

4.4.2. Effect of Biosolids Application Rate on N Availability and Leaching

High application rates of biosolids lead to high N availability in the surface soil. The extractable NH_4-N and NO_3-N seasonal pattern was similar among the three treatments. Soil NO_3-N content rapidly increased after the biosolids application due to the increased in surface soil NH_4-N content that promote nitrification (Smith et al. 1998).

As we mentioned earlier, biosolids application at the permitted rate of 225 kg PAN ha^{-1} had no effect on NO_3-N leaching. However, when biosolids were applied at the rates of 900 and 1800 kg PAN ha^{-1} we observed NO_3-N concentrations in lysimeters that may become a concern for groundwater pollution. The average NO_3-N concentration in lysimeters was 0.5 mg L^{-1}, 37.2 mg L^{-1}, and 70 mg L^{-1} in the AD225, AD900, and AD1800 treatments, respectively. Several studies observed increases in N availability and deep NO_3-N leaching associated with increasing application rates of biosolids (Brockway and Urie 1983; Egiarte et al. 2005; Hallett et al. 1999; McLaren et al. 2005; Robinson et al. 2002).

We observed that the higher application rates increased surface N content. Surface soil NO_3-N content in the AD1800 and AD900 declined by January 2006, but NO_3-N remained elevated throughout most of the study. Nitrogen concentration in soil solution in the two high application rates started to increase by the end of the growing season and remained high until the second growing season. Elevated Nitrate-N was observed for for long periods of time have been observed when biosolids were applied at a high rates due to the continue N mineralization (Brockway and Urie 1983; Egiarte et al. 2005)

N leaching is normally increased by precipitation and reduced by microbial uptake, tree and understory vegetation uptake during the growing season (Iseman et al. 1999). Once vegetation activity decreased, nitrification become more active, soil moisture content is higher, and the potential of NO_3-N leaching increased. Nitrate-N concentration in lysimeters declined to levels below the 10 mg L^{-1} by May 2007 in the AD900 and AD1800 treatments. We were not able to sample solution from lysimeters during summer due to dry soil conditions at the time of collecting the samples. Nitrate-N leaching still could occur between sample periods. Significant NO_3-N leaching has been observed when measurements were taken after storms (Egiarte et al.

2005).

Variable charge surfaces contributed to soil NO_3-N sorption capacity (Strahm and Harrison 2006). Our estimates of NO_3-N sorption capacity based on the linearized form of the Liangmur equation showed that NO_3-N sorption potential at natural soil pH was over 7000 kg ha^{-1} in the soil profile. Our results are in the range of NO_3-N sorption capacity previously observed for similar southeast soils (Eick et al. 1999). Strahm and Harrison (2007) observed a NO_3-N maximum sorption capacity of 7.5 mmol kg^{-1} for a Bt_1 horizon for a Cecil soil (fine, kaolinitic, thermic Typic Kanhapludults) collected in the Southeast Piedmont. Considering the variability associated with soil sampling, this value is similar to the estimates reported in this study (5.4 to 13.9 mmol kg^{-1}). The increased in the subsurface clayey Bt_1 and Bt_2 horizons in comparison to the Ap and BC horizons could be attributed to large clay content and surface area (Hingston 1981).

We estimated that soil maximum potential NO_3-N sorption capacity was 7159 kg ha^{-1} for the 80 cm depth soil profile. The total N applied in the three rates was 1132, 3179, and 6604 kg N ha^{-1}. The PAN approach to estimate application rates account for N mineralization and losses when biosolids are surface applied. Only a fraction of the total N applied in biosolids leach through soil (Gilmour et al. 2003). Under this assumption, we could expect little NO_3-N movement below the argillic horizon when biosolids are surface applied at the conventional rate of 225, 900, and 1800 kg PAN ha^{-1}. However, our results showed high levels of NO_3-N leaching with the two high application rates. Two things could explain this response. First, other chemical soil properties, like Al and Fe concentration, and competing anions like phosphate have a greater sorption capacity in comparison to NO_3-N (Strahm and Harrison 2007) decreasing soil NO_3-N retention. Second, forest soils tend to have permeable structure, and the presence of soil cracks

and old root channels that form preferential flow paths (Field 1989; Fisher and Binkley 1999). Franklin et al. (2007) observed that water movement through a Piedmont soil was relatively uniform in the tilled surface Ap, but preferential flow was common in the subsurface Bt horizon. The authors concluded that soil porosity was the main factor regulating water movement. Nitrate-N leachate following biosolids application to forest move through preferential flow channels and soil macropores (Sidle and Kardos 1979).

4.4.3. Effect of Season of Biosolids Application on N availability and Leaching

Our results have demonstrated that spring application significantly increased N availability and N leaching following application of biosolids to pine forest in comparison to the fall treatment. The high N availability directly affected NO_3-N concentration in lysimeters. The average NO_3-N concentration in leachate was 6.9 mg L^{-1} and 42.6 mg L^{-1}, in the fall and spring application respectively. In the spring application, NO_3-N leaching remained above 10 mg L^{-1} for a longer period of time increasing the risk of groundwater pollution. Our results agree with Evanylo (2003) that observed that N availability increased when biosolids were applied in Spring in comparison to Winter. Low temperatures likely decreased N mineralization of organic N in biosolids and nitrification of NH_4-N compared to periods with warmer temperatures (Wang et al. 2006; Wang et al. 2003). The low temperatures and precipitations after the fall application may have reduced nitrification of the fall treatments.

4.5. Conclusions

The results of this study support the idea that biosolids application at the rate of 225 kg PAN ha^{-1} could be use as a source of N in loblolly pine forest without increasing subsurface

NO_3-N leaching. Nitrogen content was variable among biosolids type. Higher levels of NH_4-N were observed in the Pellet225 treatments. In contrast, NO_3-N content was important in the AD225 and LS225 treatments. The initial inorganic N content and the N mineralization in biosolids affected N release from biosolids and time.

Despite the high soil NO_3-N sorption capacity measured, we found that higher application rates of biosolids increased NO_3-N concentration in lysimeters above water quality standards for several months. Once soil NO_3-N exceeded plant uptake, NO_3-N leaching is likely to occur through preferential flow paths generated from old roots channels and soil cracks. Substantial increases in NO_3-N concentration in subsurface soil solution following biosolids application may be a sign of potential water pollution. However, stream water pollution as a consequence of high NO_3-N concentration in groundwater will still depend on factors such NO_3-N retention in soil, NO_3-N dilution in streams, and denitrification.

We found that spring application increased soil N content in comparison to the fall application. However, NO_3-N concentration in leachate in this treatment remained elevated for a longer period of time. Based on our results, we could expect lower NO_3-N leaching concentration if biosolids were applied at the conventional rate of 225 kg PAN ha^{-1}.

Our results showed that the development of adequate rates for biosolids application to loblolly pine forest in Virginia, with low environmental impact could be higher than the 225 kg PAN ha^{-1} but below the 900 kg PAN ha^{-1}. In order to efficiently use biosolids as a source of N, without increasing groundwater pollution, future research should be focused on maximizes N release patterns with tree N uptake. The presence of understory vegetation, the accumulation of biosolids in the forest floor, and specific Piedmont forest soil characteristics, like clay content and acidity, could affect N release and movement after biosolids application.

4.6. References

Albaugh, T.J., H.L. Allen, and T.R. Fox. 2007. Historical patterns of forest fertilization in the southeastern United States from 1969 to 2004. Southern Journal of Applied Forestry 31(3):129-137.

APHA. 1998. Standard methods for examination of water and wastewater. Am Water Works Assoc., and Water Environment Federation, Washington, DC.

Aschmann, S.G., M.S. McIntosh, J.S. Angle, and R.L. Hill. 1992. Nitrogen movement under a hardwood forest amendd with liquid waste-water sludge. Agriculture Ecosystems & Environment 38(4):249-263.

Aschmann, S.G., M.S. McIntosh, J.S. Angle, R.L. Hill, and R.R. Weil. 1990. Nitrogen status of forest floor, soils, and vegetation following municipal waste-water sludge application. Journal of Environmental Quality 19(4):687-694.

Beauchamp, E.G., G.E. Kidd, and G. Thurtell. 1978. Ammonia volatilization from sewage sludge applied in field. Journal of Environmental Quality 7(1):141-146.

Beauchamp, E.G., Y.K. Soon, and J.R. Moyer. 1979. Nitrate Production from Chemically Treated Sewage Sludges in Soil. Journal of Environmental Quality 8(4):557-560.

Binkley, D., H. Burnham, and H.L. Allen. 1999. Water quality impacts of forest fertilization with nitrogen and phosphorus. Forest Ecology and Management 121(3):191-213.

Boesch, D.F., R.B. Brinsfield, and R.E. Magnien. 2001. Chesapeake Bay eutrophication: Scientific understanding, ecosystem restoration, and challenges for agriculture. P. 303-320 *in* Annual Meetings of the American-Society-of-Agronomy, Anaheim, California.

Boyle, M., and E.A. Paul. 1989. Nitrogen transformations in soils previously amended with sewage-sludge. Soil Science Society of America Journal 53(3):740-744.

Brockway, D.G. 1983. Forest Floor, Soil, and vegetation responses to sludge fertilization in Red and White-Pine Plantations. Soil Science Society of America Journal 47(4):776-784.

Brockway, D.G., and D.H. Urie. 1983. Determining Sludge Fertilization Rates for Forests from Nitrate-N in Leachate and Groundwater. Journal of Environmental Quality 12(4):487-492.

Burton, A.J., J.B. Hart, and D.H. Urie. 1990. Nitrification in slude-amended michigan forest soils. Journal of Environmental Quality 19(3):609-616.

Corey, J.C., M.W. Lower, and C.E. Davis. 1986. The sludge application program at the Savannah River plant. in The Forest Alternative for Treatment and Utilization of Municipal and Industrial Wastes. Proceedings of the Forest Lands Applications Symposium, Cole, D.W., C. Henry, and W.L. Nutter (eds.). University of Washington Press. Seattle, WA., Seattle, WA.

Donohue, S.J., and S.E. Heckendorn. 1994. Soil test recommendations for Virginia. Virginia Tech.Blacksburg, VA.

Egiarte, G., M.C. Arbestain, A. Alonsoa, E. Ruiz-Romera, and M. Pinto. 2005. Effect of repeated applications of sewage sludge on the fate of N in soils under Monterey pine stands. Forest Ecology and Management 216(1-3):257-269.

Eick, M.J., W.D. Brady, and C.K. Lynch. 1999. Charge properties and nitrate adsorption of some acid Southeastern soils. Journal of Environmental Quality 28(1):138-144.

Eldridge, S.M., K.Y. Chan, Z.H. Xu, C.R. Chen, and I. Barchia. 2008. Plant-available nitrogen supply from granulated biosolids: implications for land application guidelines. Australian Journal of Soil Research 46(5):423-436.

Evanylo, G.K. 1999a. Agricultural Land Application of Biosolids in Virginia: Risks and Concerns. Crop and Soil Environmental Sciences Publication 452-304.

Evanylo, G.K. 1999b. Agricultural Land Application of Biosolids in Virginia: Managing Biosolids for Agricultural Use. Crop and Soil Environmental Sciences Publication 452-303.

Evanylo, G.K. 2003. Effects of biosolids application timing and soil texture on nitrogen availability for corn. Communications in Soil Science and Plant Analysis 34(1-2):125-143.

Feigenbaum, S., A. Hadas, M. Sofer, and J.A.E. Molina. 1994. Clay-fixed labeled ammonium as a source of available nitrogen. Soil Science Society of America Journal 58:980-985.

Field, J.B. 1989. Water movement and chemical transport in a loblolly pine forest. *in* Georgia water resource conference, Kathryn, J. (ed.), University of Georgia, Athens, GA.

Fisher, R.F., and D. Binkley. 1999. Ecology and management of forest soils. 3rd edition. John Wiley & sons. 489 pp.

Flint, C.M., R.B. Harrison, B.D. Strahm, and A.B. Adams. 2008. Nitrogen leaching from douglas-fir forests after urea fertilization. Journal of Environmental Quality 37(5):1781-1788.

Fox, T.R., E.J. Jokela, and H.L. Allen. 2007a. The development of pine plantation silviculture in the southern United States. Journal of Forestry 105(7):337-347.

Fox, T.R., H.L. Allen, T.J. Albaugh, R. Rubilar, and C.A. Carlson. 2007b. Tree nutrition and forest fertilization of pine plantations in the southern United States. Southern Journal of Applied Forestry 31(1):5-11.

Franklin, D.H., L.T. West, D.E. Radcliffe, and P.F. Hendrix. 2007. Characteristics and genesis of preferential flow paths in a Piedmont Ultisol. Soil Science Society of America Journal 71:752-758.

Garau, M.A., M.T. Felipo, and M.C.R. Devilla. 1986. Nitrogen Mineralization of Sewage Sludges in Soils. Journal of Environmental Quality 15(3):225-228.

Gilmour, J.T. 1984. The effect of soil properties on nitrification and nitrification inhibition. Soil Science Society of America Journal 48(6):1262-1266.

Gilmour, J.T., C.G. Cogger, L.W. Jacobs, G.K. Evanylo, and D.M. Sullivan. 2003. Decomposition and plant-available nitrogen in biosolids: Laboratory studies, field studies, and computer simulation. Journal of Environmental Quality 32(4):1498-1507.

Gove, L., F.A. Nicholson, H.F. Cook, and A.J. Beck. 2002. Comparison of the effect of surface application and subsurface incorporation of enhanced treated biosolids on the leaching of heavy metals and nutrients through sand and sandy loam soils. Environmental Technology 23(2):189-198.

Gurlevik, N., D.L. Kelting, and H.L. Allen. 2004. Nitrogen mineralization following vegetation control and fertilization in a 14-year-old loblolly pine plantation. Soil Science Society of America Journal 68(1):272-281.

Hallett, R.A., W.B. Bowden, and C.T. Smith. 1999. Nitrogen dynamics in forest soils after municipal sludge additions. Water Air and Soil Pollution 112(3-4):259-278.

Henry, C., D. Sullivan, R. Rynk, K. Dorsey, and C. Cogger. 1999. Managing nitrogen from biosolids. Washington State Dept. of Ecology, [Olympia? Wash.]. 75 p.

Henry, C.L., and D.W. Cole. 1997. Use of biosolids in the forest: Technology, economics and regulations. Biomass & Bioenergy 13(4-5):269-277.

Henry, C.L., D.W. Cole, and R.B. Harrison. 1994. Use of municipal sludge to restore and improve site productivity in forestry - The pack forest sludge research program. P. 137-149 *in* IEA/BE Workshop on Ameliorative Practices for Restoring and Maintaining Long-Term Productivity in Forests, Vaxjo, Sweden.

Hingston, F.J. 1981. A review of anion adsorption. in Adsorption of inorganics at solid–liquid interfaces, Anderson, M.A., and A.J. Rubin (eds.). Ann Arbor Sciences, Ann Arbor, MI.

Howarth, R.W. 1988. Nutrient limitation of net primary production in marine ecosystems. Ann. Rev. Ecol. Sys, 19:898-1110.

Iseman, T.M., D.R. Zak, W.E. Holmes, and A.G. Merrill. 1999. Revegetation and nitrate leaching from lake states northern hardwood forests following harvest. Soil Science Society of America Journal 63:1424-1429.

Jaynes, W.F., R.E. Zartman, R.E. Sosebee, and D.B. Wester. 2003. Biosolids decomposition after surface applications in west Texas. Journal of Environmental Quality 32(5):1773-1781.

Johnson, D.W., and D.W. Cole. 1980. Anion mobility in soils: Relevance to nutrient transport from terrestrial ecosystems. Environ. Int. 3:79–90.

Jussy, J.H., M. Colin-Belgrand, E. Dambrine, J. Ranger, B. Zeller, and S. Bienaime. 2004. N deposition, N transformation and N leaching in acid forest soils. Biogeochemistry 69(2):241-262.

Kane, P.F. 2000. AOAC Methods. Association of Official Analytical Chemists.Washington, DC.

Kelty, M.J., F.D. Menalled, and M.M. Carlton. 2004. Nitrogen dynamics and red pine growth following application of pelletized biosolids in Massachusetts, USA. Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 34(7):1477-1487.

Kinjo, T., and P.F. Pratt. 1971. Nitrate adsorption: I. in some acid soils of Mexico and South America. Soil Sci. Soc. Am. Proc. 35:722–725.

Kowalenko, C.G., and S. Yu. 1996. Assessment of nitrate adsorption in soils by extraction, equilibration and column-leaching methods. Canadian Journal of Soil Science 76(1):49-57.

Littell, R., G. Milliken, W. Stroup, R. Wolfinger, and O. Schabenberger. 2006. SAS for Mixed Models, Second Edition. SAS Press, Cary, NC.

Magesan, G.N., C.D.A. McLay, and V.V. Lal. 1998. Nitrate leaching from a free-draining volcanic soil irrigated with municipal sewage effluent in New Zealand. Agriculture Ecosystems & Environment 70(2-3):181-187.

McLaren, R.G., L.M. Clucas, and M.D. Taylor. 2005. Leaching of macronutrients and metals from undisturbed soils treated with metal-spiked sewage sludge. 3. Distribution of residual metals. Australian Journal of Soil Research 43(2):159-170.

Medalie, L., W.B. Bowden, and C.T. Smith. 1994. Nutrient Leaching Following Land Application of Aerobically Digested Municipal Sewage-Sludge in a Northern Hardwood Forest. Journal of Environmental Quality 23(1):130-138.

Mehlich, A. 1953. Determination of P, Ca, Mg, K, Na, NH4. North Carolina Dept. of Agriculture, Agronomic Division.

Powers, R.F. 1980. Mineralizable soil-nitrogen as an index of nitrogen availability to forest trees. Soil Science Society of America Journal 44(6):1314-1320.

Prescott, C.E., and L.M. Zabek. 1997. Growth response and nutrient availability in western redcedar plantations following amendment with fish-wood compost and straw. Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 27(4):598-602.

Quemada, M., B. Lassa, C. Lamsfus, and P.M. Aparicio Tejo. 1998. Ammonia Volatilization from Surface or Incorporated Biosolids by the Addition of Dicyandiamide. Journal of Environmental Quality 27:980-983.

Riekerk, H. 1981. Effects of sludge disposal on drainage solutions of 2 forest soils. Forest Science 27(4):792-800.

Robinson, M.B., and P.J. Polglase. 2000. Volatilization of nitrogen from dewatered biosolids. Journal of Environmental Quality 29(4):1351-1355.

Robinson, M.B., P.J. Polglase, and C.J. Weston. 2002. Loss of mass and nitrogen from biosolids applied to a pine plantation. Australian Journal of Soil Research 40(6):1027-1039.

Rowell, D.M., C.E. Prescott, and C.M. Preston. 2001. Decomposition and nitrogen mineralization from biosolids and other organic materials: Relationship with initial chemistry. Journal of Environmental Quality 30(4):1401-1410.

Sidle, R.C., and L.T. Kardos. 1979. Nitrate leaching in a sludge-treated forest soil. Soil Science Society of America Journal 43(2):278-282.

Silva, R.G., S.M. Holub, E.E. Jorgensen, and A.N.M. Ashanuzzaman. 2005. Indicators of nitrate leaching loss under different land use of clayey and sandy soils in southeastern Oklahoma. Agriculture Ecosystems & Environment 109(3-4):346-359.

Simmons, J.A., J.B. Yavitt, and T.J. Fahey. 1996. Watershed liming effects on the forest floor N cycle. Biogeochemistry 32(3):221-244.

Smith, S.R., V. Woods, and T.D. Evans. 1998. Nitrate dynamics in biosolids-treated soils. I. Influence of biosolids type and soil type. Bioresource Technology 66:139-149.

Sommers, L.E. 1977. Chemical composition of sewage sludges and analysis of their potential use as fertilizers. Journal of Environmental Quality 6(2):225-232.

Sommers, L.E., D.W. Nelson, and D.J. Silviera. 1979. Transformations of carbon, nitrogen, and metals in soils treated with waste materials. Journal of Environmental Quality 8(3):287-294.

Strahm, B.D., and R.B. Harrison. 2006. Nitrate sorption in a variable-charge forest soil of the Pacific Northwest. Soil Science 171(4):313-321.

Strahm, B.D., and R.B. Harrison. 2007. Mineral and organic matter controls on the sorption of macronutrient anions in variable-charge soils. Soil Science Society of America Journal 71(6):1926-1933.

Strahm, B.D., and R.B. Harrison. 2008. Controls on the Sorption, Desorption, and Mineralization of Low-Molecular-Weight Organic Acids in Variable-Charge Soils. Soil Science Society of America Journal 72(6):1653-1664.

Terry, R.E., D.W. Nelson, and L.E. Sommers. 1979. Decomposition of anaerobically digested sewage sludge as affected by soil environmental-conditions. Journal of Environmental Quality 8(3):342-347.

Terry, R.E., D.W. Nelson, and L.E. Sommers. 1981. Nitrogen transformation in sewage sludge amended soils as affected by soil environmental factors. Soil Science Society of America Journal 45(3):506-513.

USEPA. 1983. Methods for the chemical analysis of water and wastes (MCAWW). EPA/600/4-79/020. NTIS item PB84-128677. in Environ. Monitoring and Support Lab. Office of Res. and Dev., USEPA, Cincinnati, OH.

USEPA. 1986. Quality criteria for water EPA-440/5-86-001. U.S. Environmental Protection Agency.Washington, DC.

USEPA. 1995. Process design for agricultural utilization. In Process Design Manual–Land Application of Sewage Sludge and Domestic Septage EPA/625/R-95/001; U.S. Environmental Protection Agency, Washington, DC.

USEPA. 2002. Test methods for evualating solid waste, physical/chemical methods SW-846 manual U.S. U.S. Gov Print Office. U.S. Environmental Protection Agency, Washington, DC.

Wang, H., M.O. Kimberley, G.N. Magesan, R.B. McKinley, J.R. Lee, J.M. Lavery, P.D.F. Hodgkiss, T.W. Payn, P.J. Wilks, C.R. Fisher, and D.L. McConchie. 2006. Midrotation effects of biosolids application on tree growth and wood properties in a Pinus radiata plantation. Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 36(8):1921-1930.

Wang, H.L., G.N. Magesan, M.O. Kimberley, T.W. Payn, P.J. Wilks, and C.R. Fisher. 2004a. Environmental and nutritional responses of a Pinus radiata plantation to biosolids application. Plant and Soil 267(1-2):255-262.

Wang, H., G.N. Magesan, P.W. Clinton, and J.M. Lavery. 2004b. Using natural N-15 abundances to trace the fate of waste-derived nitrogen in forest ecosystems: New Zealand case studies. P. 31-38 in 4th International Conference on Applications of Stable Isotope Techniques to Ecological Studies, Wellington, New Zealand.

Wang, H.L., S.L. Brown, G.N. Magesan, A.H. Slade, M. Quintern, P.W. Clinton, and T.W. Payn. 2008. Technological options for the management of biosolids. Environmental Science and Pollution Research 15(4):308-317.

Wang, H.L., M.O. Kimberley, and M. Schlegelmilch. 2003. Biosolids-derived nitrogen mineralization and transformation in forest soils. Journal of Environmental Quality 32(5):1851-1856.

Wells, C.G., C.E. Murphy, C. Davis, D.M. Stone, and G.J. Hollod. 1985. Effect of sewage sludge from two sources on element flux in soil solution of loblolly pine plantations. P. 154±165. in The Forest Alternative for Treatment and Utilization of Municipal and Industrial Wastes, Cole, D.W., C. Henry, and W.L. Nutter (eds.). University of Washington Press, Seattle, WA.

4.7. Tables and Figures

Table 4.1. Selected chemical and physical properties from the mineral soil at four different depths, from a 17-year-old loblolly pine plantation, Amelia County, VA.

Soil Depth (cm)	PH (1:1)	BD g cc^{-1}	C	N	P	K	Ca	Mg
			mg Kg^{-1}					
0-20	5.47	1.24	5700	484.0	4.0	32.0	342.0	73.0
20-40	6.18	1.39	2476	174.5	2.0	34.5	373.8	128.8
40-60	5.86	1.49	1695	165.8	2.0	31.5	334.5	129.8
60-80	5.44	1.54	1135	96.8	2.0	32.0	248.3	112.5

Table 4.2. Selected properties for biosolids surface applied in a 17-year-old loblolly pine plantation in Amelia County, VA. Biosolids source for the fall application treatment was Alexandria, VA. Biosolids sources for the spring application were Blue Plain (DC) and Back River, MD, and Baltimore, MD.

Properties	Lime stabalized (Blue Plain)	Anaerobically Digested (Alexandria)	Anaerobically Digested (Back River)	Pelletized (Baltimore)
pH	12.3	8.1	8.2	5.6
		mg kg^{-1}		
Solids	352300	241500	205100	929500
Nitrogen (TKN)	31300	47500	50300	56600
Water Insol N	-	-	-	50900
Ammonia-N	1100	10200	11800	600
Phosphorus	10600	17300	20200	16100
Potassium	1300	1100	2100	2700
Sulfur	4300	10400	9300	5500
Calcium	114300	29400	22400	11200
Magnesium	2300	3300	3800	2200
Sodium	200	500	1000	400
Iron	44693	50749	55100	13682
Manganese	197	946	793	159
Copper	158	403	463	261
Zinc	314	796	867	395
Cadmium	1.2	5.8	10	-
Chromium	38	76	75	89
Nickel	16	37	36	14
Lead	38	66	66	20
Arsenic	2.9	3.6	2.1	3.9
Mercury	0.3	1.4	1.04	0.4
Selenium	2.0	3.6	4.7	2.1

Table 4.3. Final application rates and nutrient applied after of surface application of biosolids at a 17-year-old loblolly pine plantation in Amelia County, VA. Fall treatment application was anaerobically digested biosolids at a rate of 900 kg PAN ha^{-1}. Spring treatments applications were lime stabilized, pelletized, urea+DAP, and anaerobically digested at a rate of 225 kg PAN ha^{-1}, and control. Anaerobically digested biosolids was also added at a rate of 900 kg PAN ha^{-1} and 1800 kg PAN ha^{-1}.

Treatments	Treatment name	Target PAN	Actual PAN	Total N	N-Org	Total P	Total K	Total Ca	Dry Weight
					Kg ha^{-1}				Mg ha^{-1}
Fall Application									
Anaer. Digested	AD900F	900	847	3202	2514	1166	74	1982	67.4
Spring Application									
Lime Stabilized	LS225	225	275	920	888	312	38	3360	29.4
Pellets	Pellet225	225	306	306	291	84	14	58	5.2
Urea + DAP	U+DAP225	225	214	214	0	23	-	-	-
Anaer. Digested	AD225	225	306	1132	866	455	47	504	22.5
Anaer. Digested	AD900S	900	860	3179	2433	1277	133	1416	63.2
Anaer. Digested	AD1800	1800	1786	6604	5055	2652	276	2941	131.3

Table 4.4. Summary of Anova for the effect of biosolids and sampling time on KCl extractable NH_4, KCl extractable NO_3, leachate NH_4, and leachate NO_3 after surface application of biosolids in the 17-year-old loblolly pine plantation in Amelia County, VA.

	Extractable KCl-N					Leaching				
	Num	NH_4		NO_3		Num	NH_4		NO_3	
Effect	Df	F value	Pr >F	F value	Pr >F	Df	F value	Pr >F	F value	Pr >F
Block	3	0.23	0.8744	0.98	0.4005	3	1.04	0.376	0.61	0.6089
Treatment	7	70.9	<.0001	94.86	<.0001	7	28.24	<.0001	106.99	<.0001
Time	20	32.99	<.0001	44.71	<.0001	14	2.15	0.0102	18.25	<.0001
Treatment *Time	140	4.66	<.0001	3.83	<.0001	98	3.48	<.0001	3.15	<.0001

Table 4.5. Contrast for KCl extractable NH_4-N, KCl extractable NO_3-N, NH_4-N leaching, and NO_3-N leaching means average over 21 months in a17-year-old loblolly pine plantation following biosolids surface application of biosolids Treatments were anaerobically digested (AD225, AD900F, AD900S, AD1800), lime stabilized (LS225), pelletized (Pellet225) biosolids, urea+DAP, and control.

Contrast	KCl-NH_4	KCl-NO_3	L-NH_4	L-NO_3
		Pr > F		
Type of biosolids				
Control vs LS225	<.0001	<.0001	0.5092	0.0036
Control vs AD225	<.0001	<.0001	0.1642	0.1102
Control vs Pellet225	<.0001	<.0001	0.1081	0.0002
Control vs U+DAP225	<.0001	<.0001	0.4579	0.2201
AD225 vs U+DAP225	0.005	0.1965	0.5178	0.7137
LS225 vs AD225	0.4504	0.8055	0.4509	0.1882
LS225 vs Pellet225	<.0001	0.0509	0.3317	0.408
LS225 vs U+DAP225	0.0004	0.2915	0.9244	0.0932
Pellet225 vs AD225	<.0001	0.0283	0.8318	0.0345
Pellet225 vs U+DAP225	<.0001	0.3973	0.3894	0.0136
Application rate				
AD1800 vs AD900	0.0511	0.7919	0.8171	0.2329
AD1800 vs AD225	<.0001	<.0001	<.0001	<.0001
AD900 vs AD225	<.0001	<.0001	<.0001	<.0001
Season of application				
AD900F vs AD900S	0.0003	<.0001	<.0001	<.0001

Table 4.6. Estimated KCl-NH_4 means at each sampling date following surface application of biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County VA at the rate of 225 kg PAN ha^{-1}. Treatments were control, anaerobically digested (AD225), lime stabilized (LS225), pelletized (Pellet225) biosolids, and urea + DAP. Units are kg NH_4 per ha. Different letters at each month indicate significant difference between treatments (α<0.05).

Month	Control	AD225	LS225	Pellet225	U+DAP225
	KCl-NH_4 (kg ha^{-1})				
Feb-06	3.2	3.9	5.0	4.3	6.1
Mar-06	6.7 b	20.1 a	8.9 b	25.1 a	34.4 a
Apr-06	8.2 b	12.9 b	10.1 b	28.0 a	26.2 a
May-06	10.7 c	24.7 b	10.4 c	33.4 a	60.9 a
Jun-06	10.9 c	11.2 c	7.5 c	38.3 a	25.8 b
Jul-06	9.7 c	8.9 c	21.5 b	87.8 a	19.1 b
Aug-06	12.1 b	16.0 b	11.4 b	54.6 a	12.8 b
Sep-06	7.8 c	10.5 bc	15.9 b	54.7 a	12.3 bc
Oct-06	11.4	13.3	14.6	11.3	8.3
Nov-06	10.2	7.1	9.4	9.1	7.5
Dec-06	2.8 b	7.7 a	5.8 a	12.8 a	4.6 ab
Jan-07	4.2 b	9.4 a	8.5 ab	13.5 a	11.6 a
Feb-07	6.2 a	9.4 a	2.6 b	8.5 a	9.1 a
Mar-07	5.1	6.2	7.6	6.8	7.8
Apr-07	5.0 b	10.3 b	7.3 b	19.8 a	9.5 b
May-07	6.1 b	9.3 ab	13.7 a	11.5 ab	7.8 ab
Jun-07	9.2 ab	9.5 ab	16.3 a	15.7 ab	8.0 b
Jul-07	9.5 b	11.6 b	13.0 b	23.9 a	11.3 b
Aug-07	7.3 b	11.5 ab	13.2 a	19.6 a	16.9 a
Sep-07	7.7 ab	7.7 ab	7.1 b	7.9 ab	14.2 a
Oct-07	5.3	6.0	10.9	8.5	7.8

Table 4.7. Estimated KCl-NO_3 means at each sampling date following surface application of biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County VA at the rate of 225 kg PAN ha^{-1}. Treatments were control, anaerobically digested (AD225), lime stabilized (LS225), pelletized (Pellet225) biosolids, and urea + DAP. Units are kg NO_3 per ha. Different letters at each month indicate significant difference between treatments (α<0.05).

Month	Control	AD 225	LS 225	Pellet225	U+DAP 225
	KCl-NO_3 (kg ha^{-1})				
Feb-06	1.41	0.97	1.33	0.51	0.54
Mar-06	2.04 a	2.40 a	1.62 ab	1.90 ab	1.01 b
Apr-06	2.01 b	4.65 a	4.42 ab	5.37 a	3.99 ab
May-06	2.50 c	10.75 a	4.82 bc	9.09 ab	9.07 ab
Jun-06	0.91 c	8.81 ab	9.24 ab	5.67 b	14.51 a
Jul-06	1.24 d	7.66 bc	11.09 b	4.38 c	29.34 a
Aug-06	3.98 c	14.31 ab	24.04 a	8.17 bc	18.51 ab
Sep-06	5.18 b	19.24 a	9.09 ab	14.12 a	9.70 ab
Oct-06	3.67 b	13.39 a	19.76 a	15.07 a	13.66 a
Nov-06	2.77 c	7.01 ab	7.70 ab	12.77 a	3.80 b
Dec-06	0.89 b	3.00 a	5.69 a	2.65 a	2.64 a
Jan-07	2.40	3.06	3.03	2.35	2.19
Feb-07	2.86	3.37	2.92	3.58	2.99
Mar-07	1.51 c	7.56 a	4.41 ab	2.96 ab	2.12 bc
Apr-07	2.84 c	12.44 a	3.64 c	4.58 bc	10.27 ab
May-07	4.27	7.50	10.50	5.62	4.96
Jun-07	1.57 b	8.01 a	11.34 a	5.13 a	8.19 a
Jul-07	1.73 b	7.77 a	6.24 a	8.22 a	6.07 a
Aug-07	3.66 b	5.80 ab	7.11 ab	9.86 a	5.59 ab
Sep-07	2.14 b	5.05 a	7.41 a	5.25 a	6.74 a
Oct-07	2.41	4.17	5.33	4.36	5.48

Table 4.8. Estimated means at each sampling date of NH_4-N concentration in lysimeters at 80 cm depth following surface application of biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County, VA. Application rate was 225 kg PAN ha^{-1}. Treatments were control, anaerobically digested (AD225), lime stabilized (LS225), pelletized (Pellet225) biosolids, and urea + DAP. Different letters at each month indicate significant difference between treatments ($\alpha<0.05$). Missing values indicated no sample collection due to soil drought.

Month	Control	AD 225	LS 225	Pellets 225	U+DAP 225
			NH_4-N (mg L^{-1})		
Feb-06	0.15	0.18	0.16	0.16	0.14
Mar-06	0.08	0.19	0.16	0.09	0.15
Apr-06	0.17	0.20	0.12	0.19	0.09
May-06					
Jun-06					
Jul-06					
Aug-06	0.05	0.09	0.16	0.15	0.42
Sep-06	0.10	0.20	0.16	0.30	0.34
Oct-06	0.13	0.13	0.22	0.20	0.14
Nov-06	0.18	0.57	0.27	0.30	0.09
Dec-06	0.14	0.14	0.30	0.20	0.13
Jan-07	0.19	0.46	0.19	0.26	0.31
Feb-07	0.19	0.20	0.12	0.12	0.17
Mar-07	0.19	0.22	0.18	0.33	0.17
Apr-07	0.19	0.19	0.31	0.45	0.14
May-07	0.17	0.21	0.09	0.17	0.15
Jun-07					
Jul-07					
Aug-07					
Sep-07	0.17	0.18	0.15	0.53	0.19
Oct-07	0.19	0.19	0.18	0.14	0.16

Table 4.9. Estimated means at each sampling date for NO_3-N concentration in lysimeters at 80 cm depth following surface application of biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County VA. Application rate was 225 kg ha^{-1} PAN. Treatments were control, anaerobically digested (AD225), lime stabilized (LS225), pelletized (Pellet225) biosolids, and urea + DAP. Different letters at each month indicate significant difference between treatments (α<0.05). Missing values indicated no sample collection due to soil drought.

Month	Control	AD 225	LS 225	Pellets 225	U+DAP 225
	NO_3-N (mg L^{-1})				
Feb-06	0.27	0.17	0.20	0.31	0.22
Mar-06	0.37	0.26	0.45	0.19	0.22
Apr-06	0.25	0.33	0.36	0.27	0.22
May-06					
Jun-06					
Jul-06					
Aug-06	0.47	1.04	1.36	1.63	0.43
Sep-06	0.12 b	0.63 ab	0.99 a	2.48 a	0.72 a
Oct-06	0.42 b	1.19 ab	0.86 ab	1.98 a	1.89 a
Nov-06	0.22 b	0.87 ab	1.08 ab	3.18 a	0.37 ab
Dec-06	0.22 ab	0.40 ab	0.99 a	1.02 a	0.12 b
Jan-07	0.31	0.33	0.96	0.70	0.22
Feb-07	0.18	0.64	0.28	0.71	0.75
Mar-07	0.39 ab	1.11 ab	0.36 ab	0.59 a	0.15 b
Apr-07	0.25 b	0.36 ab	0.35 ab	0.98 a	0.44 ab
May-07	0.13	0.09	0.28	0.41	0.52
Jun-07					
Jul-07					
Aug-07					
Sep-07	0.17	0.09	0.32	0.35	0.09
Oct-07	0.13	0.11	0.21	0.37	0.17

Table 4.10. Estimated means at each sampling date for KCl-NH_4 after surface application of anaerobically digested biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County VA. Application rates were 225 (AD225), 900 (AD900), and 1800 (1800) kg PAN ha^{-1} . Different letters at each month indicate significant difference between treatments (α<0.05).

Month	AD 225	AD 900	AD 1800
	KCl-NH_4 (kg ha^{-1})		
Feb-06	3.9	3.6	4.0
Mar-06	20.1 b	41.7 a	58.7 a
Apr-06	12.9 b	28.6 a	44.5 a
May-06	24.7 b	56.9 a	95.4 a
Jun-06	11.2 b	60.2 a	108.8 a
July-06	8.9 b	27.3 a	50.4 a
Aug-06	16.0 b	63.5 a	73.8 a
Sep-06	10.5 c	25.6 b	83.4 a
Oct-06	13.3 b	24.4 a	33.3 a
Nov-06	7.1 b	25.7 a	19.7 a
Dec-06	7.7 b	26.6 a	15.2 a
Jan-07	9.4 b	18.9 a	9.9 ab
Feb-07	9.4	11.3	14.6
Mar-07	6.2 b	7.1 b	17.6 a
Apr-07	10.3	12.5	14.3
May-07	9.3 b	19.1 a	11.2 ab
Jun-07	9.5 b	20.8 a	21.3 a
Jul-07	11.6 b	49.8 a	19.5 b
Aug-07	11.5	14.1	22.4
Sep-07	7.7	13.8	11.3
Oct-07	6.0	9.4	9.1

Table 4.11. Estimated means at each sampling date for KCl-NO_3 after surface application of anaerobically digested biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County VA. Application rates were 225 (AD225), 900 (AD900), and 1800 (AD1800) kg PAN ha^{-1} . Different letters at each month indicate significant difference between treatments (α<0.05).

Month	AD 225	AD 900	AD 1800
	KCl-NO_3 (kg ha^{-1})		
Feb-06	1.0	1.5	0.7
Mar-06	2.4	4.2	3.7
Apr-06	4.6	8.2	6.3
May-06	10.7	14.5	19.0
Jun-06	8.8 b	28.9 a	50.8 a
Jul-06	7.7 b	37.8 a	30.6 a
Aug-06	14.3 b	39.1 a	55.7 a
Sep-06	19.2 b	62.7 a	89.0 a
Oct-06	13.4 b	47.4 a	80.5 a
Nov-06	7.0 b	30.8 a	32.3 a
Dec-06	3.0 b	20.8 a	13.9 a
Jan-07	3.1 b	21.3 a	13.5 a
Feb-07	3.4 b	17.5 a	12.3 a
Mar-07	7.6 b	23.0 a	24.2 a
Apr-07	12.4 ab	9.5 b	17.8 a
May-07	7.5	10.4	6.1
Jun-07	8.0	7.6	8.8
Jul-07	7.8	4.0	9.4
Aug-07	5.8 b	14.5 a	12.9 ab
Sep-07	5.0 b	11.4 a	4.8 b
Oct-07	4.2 b	9.2 a	6.0 ab

Table 4.12. Estimated means at each sampling date for NH_4-N concentration in lysimeters at 80 cm soil depth after surface application of anaerobically digested biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County VA. Application rates were 225 (AD225), 900 (AD900), and 1800 (AD1800) kg PAN ha^{-1}. Different letters at each month indicate significant difference between treatments (α<0.05). Missing values indicated no sample collection due to soil drought.

Month	AD 225	AD 900	AD 1800
	NH_4-N (mg L^{-1})		
Feb-06	0.18	0.20	0.07
Mar-06	0.19	0.16	0.70
Apr-06	0.20	2.86	0.90
May-06			
Jun-06			
Jul-06			
Aug-06	0.09 b	0.72 a	1.41 a
Sep-06	0.20 b	1.00 a	2.13 a
Oct-06	0.13 b	0.54 b	2.00 a
Nov-06	0.57 b	2.01 a	1.15 a
Dec-06	0.14 b	0.76 ab	1.07 a
Jan-07	0.46	1.02	0.46
Feb-07	0.20	0.36	0.72
Mar-07	0.22	0.36	0.25
Apr-07	0.19	0.15	0.51
May-07	0.21	0.37	0.82
Jun-07			
Jul-07			
Aug-07			
Sep-07	0.18 ab	0.89 a	0.15
Oct-07	0.19	0.17	0.50

Table 4.13. Estimated means at each sampling date for NO_3-N concentration in lysimeters at 80 cm soil depth after surface application of anaerobically digested biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County, VA. Application rates were 225 (AD225), 900 (AD900), and 1800 (AD1800) kg PAN ha^{-1}. Different letters at each month indicate significant difference between treatments (α<0.05). Missing values indicated no sample collection due to soil drought.

Month	AD225	AD900	AD1800
	NO_3-N(mg L^{-1})		
Feb-06	0.17	0.13	0.24
Mar-06	0.26 b	1.92 a	1.03 ab
Apr-06	0.33	0.60	0.53
May-06			
Jun-06			
Jul-06			
Aug-06	1.04 b	29.97 a	133.64 a
Sep-06	0.63 b	62.94 a	179.80 a
Oct-06	1.19 b	57.14 a	127.08 a
Nov-06	0.87 b	97.79 a	103.97 a
Dec-06	0.40 b	69.20 a	88.72 a
Jan-07	0.33 b	43.39 a	159.39 a
Feb-07	0.64 b	68.67 a	90.73 a
Mar-07	1.11 b	81.15 a	81.82 a
Apr-07	0.36 b	34.89 a	70.99 a
May-07	0.09 b	5.77 a	5.91 a
Jun-07			
Jul-07			
Aug-07			
Sep-07	0.09 b	2.02 a	2.71 a
Oct-07	0.11 b	5.68 a	3.42 a

Table 4.14. Estimated means since treatment application for KCl extractable NH_4 after surface application of anaerobically digested biosolids in November 2005 (fall), and March 2006 (spring) to a 17-year-old loblolly pine plantation in Amelia County, VA. Application rate was 900 kg PAN ha^{-1}. Different letters at each month indicate significant difference between treatments (α<0.05).

Month since Application	AD900F	AD900S
	KCl-NH_4 (kg ha^{-1})	
1	66.5 a	3.6 b
2	77.2	41.7
3	23.0	28.6
4	36.7	56.9
5	28.0 b	60.2 a
6	30.1	27.3
7	31.7 b	63.5 a
8	22.1	25.6
9	11.7 b	24.4 a
10	25.1	25.7
11	20.1	26.6
12	13.2	18.9
13	18.0	11.3
14	13.0	7.1
15	5.2 b	12.5 a
16	4.3 b	19.1 a
17	3.7 b	20.8 a
18	6.9 b	49.8 a
19	6.3 b	14.1 a
20	14.2	13.8
21	20.8 a	9.4 b

Table 4.15. Estimated means since treatment application for KCl extractable NO_3 after surface application of anaerobically digested biosolids in November 2005 (fall), and March 2006 (spring) to a 17-year-old loblolly pine plantation in Amelia County, VA. Application rate was 900 kg PAN ha^{-1}. Different letters at each month indicate significant difference between treatments ($\alpha<0.05$).

Month since Application	AD900F	AD900S
	KCl-NO_3 (kg ha^{-1})	
1	1.4	1.5
2	0.7	4.2
3	4.1	8.2
4	2.6	14.5
5	10.0 b	28.9 a
6	22.2	37.8
7	21.4 b	39.1 a
8	30.7 b	62.7 a
9	16.8	47.4
10	39.2	30.8
11	38.5	20.8
12	47.8 a	21.3 b
13	20.6	17.5
14	10.9	23.0
15	7.9	9.5
16	4.2	10.4
17	5.3	7.6
18	7.1	4.0
19	12.5	14.5
20	9.5	11.4
21	5.9	9.2

Table 4.16. Estimated means since treatment application for NH_4-N n soil solution in lysimeters at 80 cm depth after surface application of anaerobically digested biosolids in November 2005 (fall), and March 2006 (spring) to a 17-year-old loblolly pine plantation in Amelia County, VA. Application rate was 900 kg PAN ha^{-1}. Missing values indicated no sample collection due to soil drought.

Month since Application	AD900F	AD900S
	NH_4-N (mg L^{-1})	
1	30.19	0.20
2	39.17	0.16
3	19.15	2.86
4	14.17	
5	6.17	
6	1.07	
7		0.72
8		1.00
9		0.54
10	0.96	2.01
11	0.54	0.76
12	1.21	1.02
13	0.95	0.36
14	0.40	0.36
15	0.44	0.15
16	0.11	0.37
17	0.18	
18	0.22	
19	0.55	
20		0.89
21		0.17

Table 4.17. Estimated means for NO_3-N in soil solution in lysimeters at 80 cm depth after surface application of anaerobically digested biosolids on November 2005 (fall), and March 2006 (spring) to a 17-year-old loblolly pine plantation in Amelia County, VA. Application rate was 900 kg PAN ha^{-1}. Missing values indicated no sample collection due to soil drought.

Month since Application	AD900F	AD900S
	NO_3-N(mg L^{-1})	
1	0.17	0.13
2	0.50	1.92
3	0.58	0.60
4	0.81	
5	1.10	
6	1.42	
7		29.97
8		62.94
9		57.14
10	42.57	97.79
11	15.10	69.20
12	11.53	43.39
13	9.63	68.67
14	7.81	81.15
15	10.70	34.89
16	3.05	5.77
17	2.61	
18	1.65	
19	0.64	
20		2.02
21		5.68

Table 4.18. Langmuir isotherm equation parameters for the theoretical maximum quantity nitrate-N sorbed (Nmax) and the sorptive affinity (b), and for mineral soil collected at four different depths in a 17-year-old loblolly pine plantation.

	NO_3^-N max		b
Soil Depth	**mmol kg soil^{-1}**	**kg ha^{-1}**	**mmol kg soil^{-1}**
Ap (0 to 20 cm)	10.31	1582	0.0921
Bt_1 (20 to 40 cm)	11.52	1985	0.1126
Bt_2 (40 to 60 cm)	13.92	2571	0.1122
BC (60 to 80 cm)	5.35	1021	0.3713
Total	41.1	7159	-------

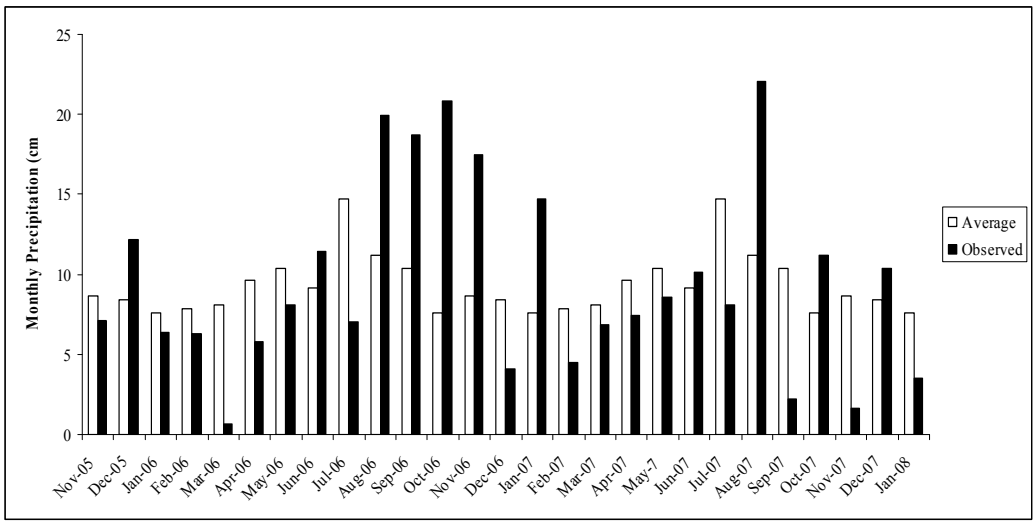


Figure 4.1. Average precipitation from 15-year period, (Blackstone, VA) and observed total monthly precipitation during the study duration (Amelia County, VA).

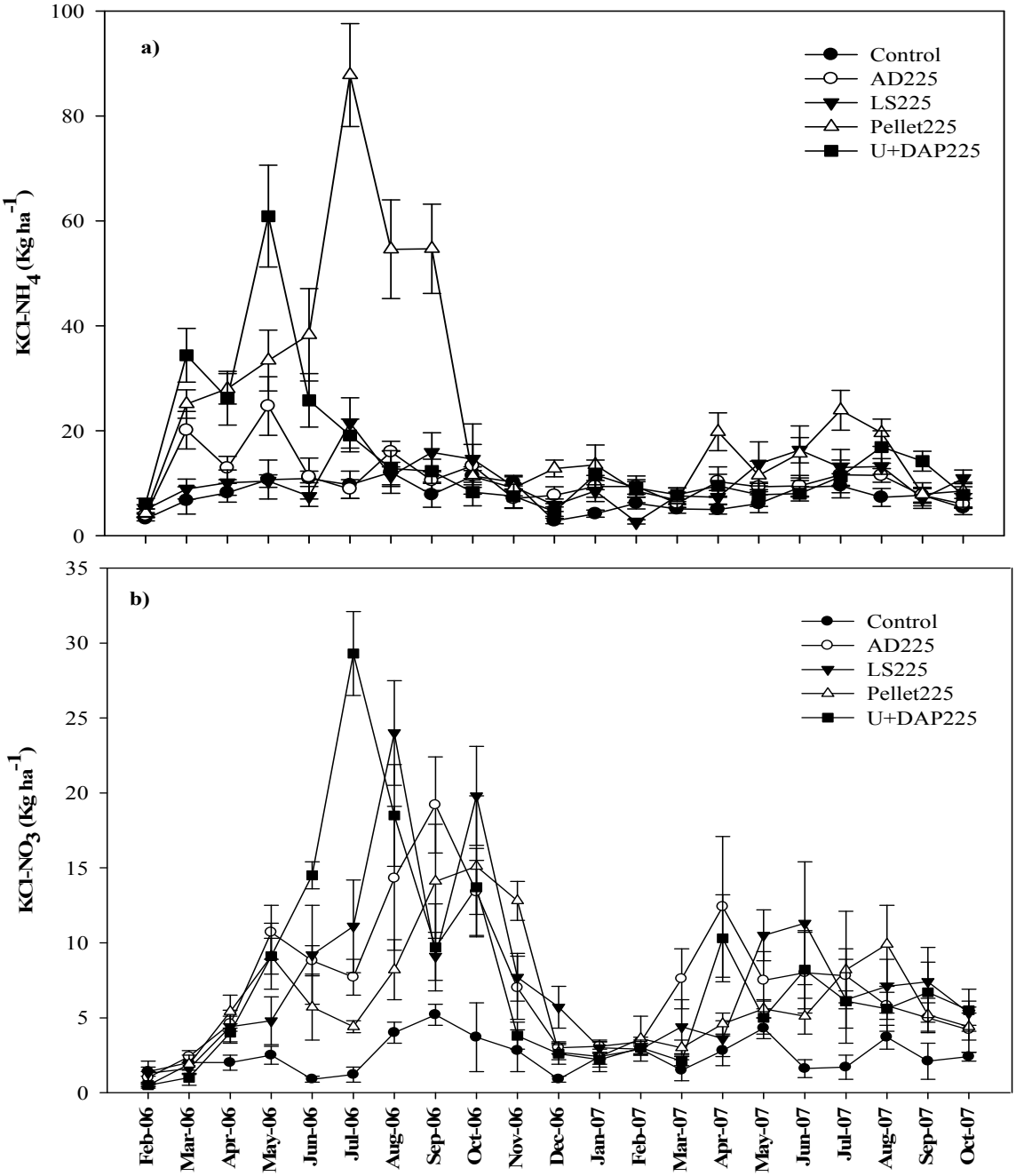


Figure 4.2. KCl extractable NH_4 (a), and KCl extractable NO_3 (b) from the surface 15 cm mineral soil following application of biosolids in a loblolly pine plantation. Treatments were anaerobically digested (AD225), lime stabilized (LS225), pelletized biosolids (Pellet225), urea+DAP, and control. Treatments were applied during March of 2006 at the rate of 225 PAN kg ha^{-1}. Graphs scales are different. Brackets indicate ± SE.

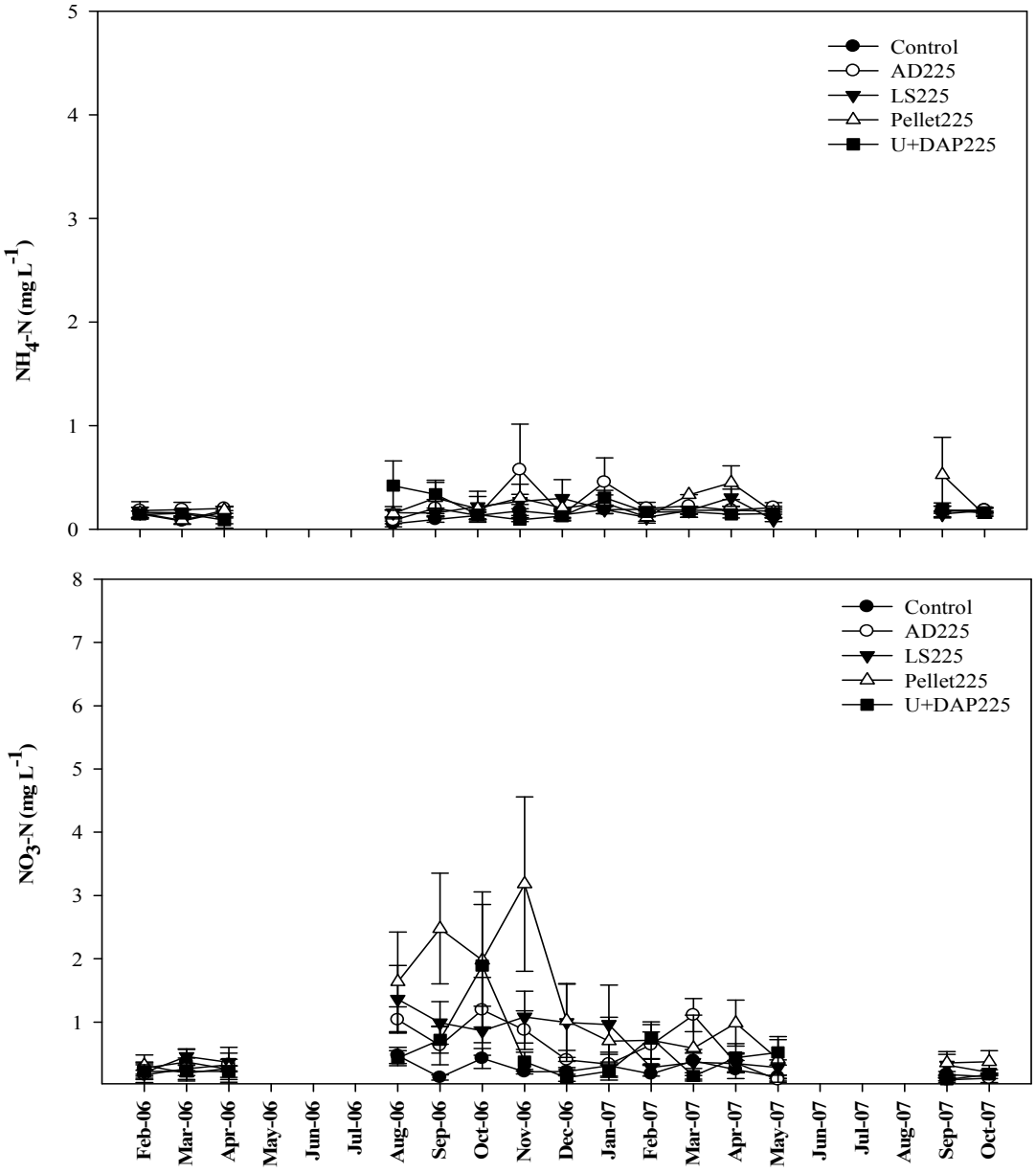


Figure 4.3.NH_4-N (a), and NO_3-N (b) concentration in soil solution from lysimeters at each sampling date after surface application of biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County VA. Application rate was 225 kg PAN ha^{-1} . Treatments were control, anaerobically digested (AD225), lime stabilized (LS225), pelletized (Pellet225) biosolids, and U+DAP. Missing values indicate no soil solution collected from lysimeters. Graphs scales are different. Brackets indicate ± SE.

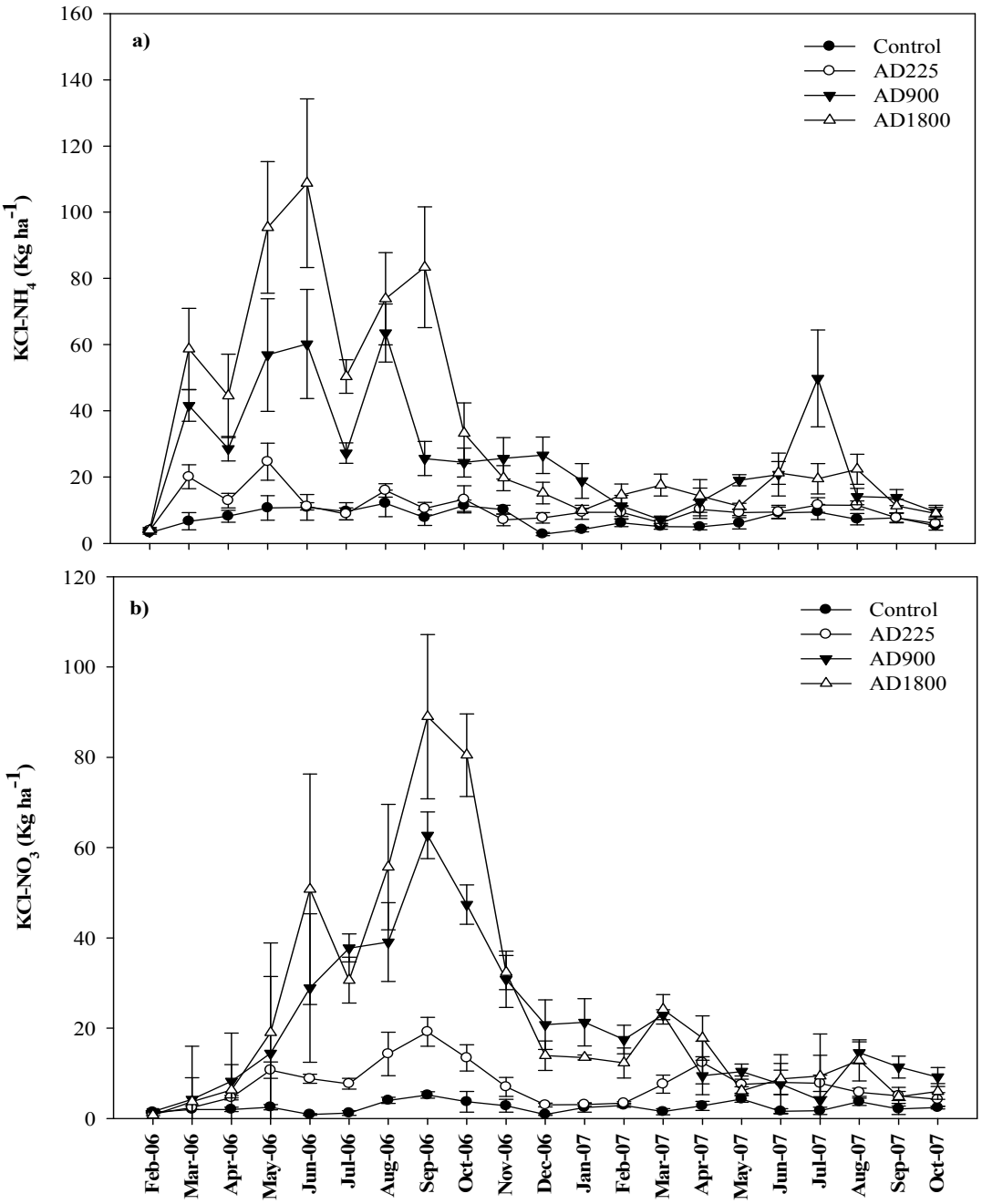


Figure 4.4. KCl extractable NH_4 (a), and KCl extractable NO_3 (b) from the surface 15 cm mineral soil following application of biosolids in a loblolly pine plantation. Treatments were control, and anaerobically digested biosolids applied during March 2006 at the rates of 225 (AD225), 900 (AD900), and 1800 (AD1800) kg PAN ha^{-1}. Graphs scales are different. Brackets indicate ± SE.

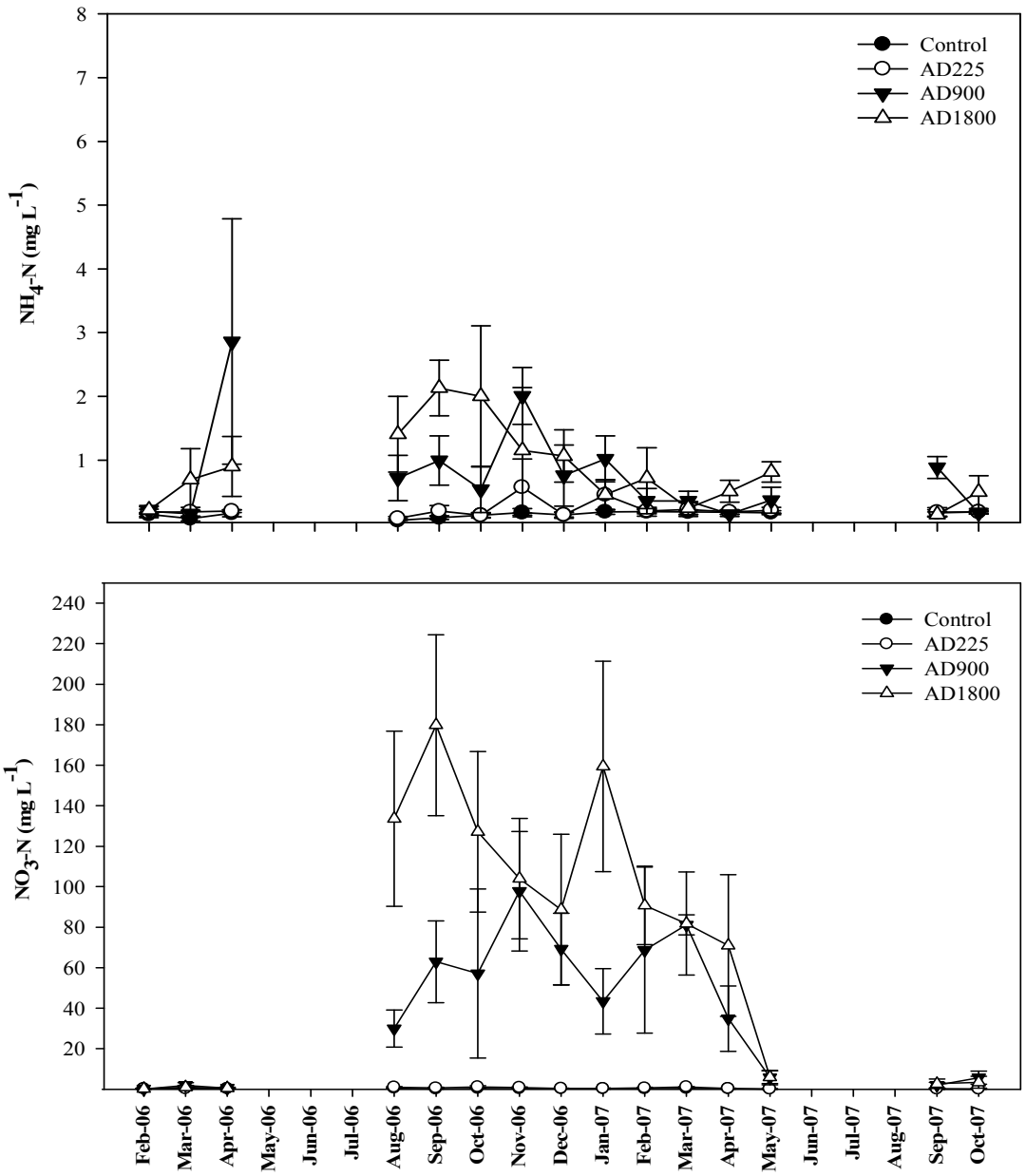


Figure 4.5. NH_4-N (a), and NO_3-N (b) concentration in soil solution from lysimeters following surface application of biosolids on March 2006 to a 17-year-old loblolly pine plantation in Amelia County, VA. Treatments were control, and anaerobically digested biosolids applied during March 2006 at the rates of 225 (AD225), 900 (AD900), and 1800 (AD1800) kg PAN ha^{-1}. Scale units are different. Brackets indicate ± SE.

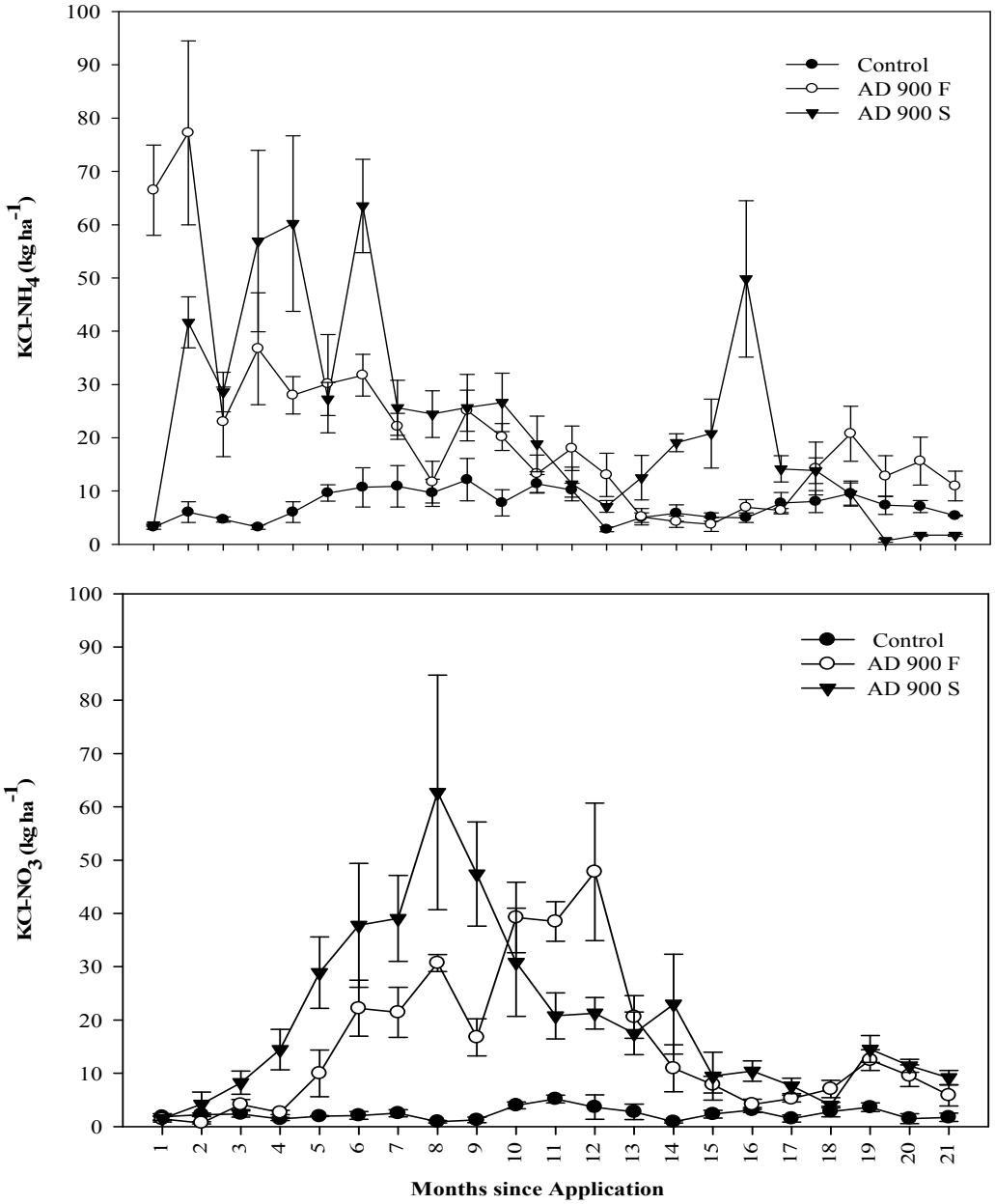


Figure 4.6. KCl extractable NH_4 (a), and KCl extractable NO_3 (b) content in the surface 15 cm mineral soil over time following biosolids application in a loblolly pine plantation. Treatments were control, and anaerobically digested biosolids applied during November 2005 (fall), and March 2006 (spring) at the rate of 900 kg PAN ha^{-1}. Brackets indicate ± SE.

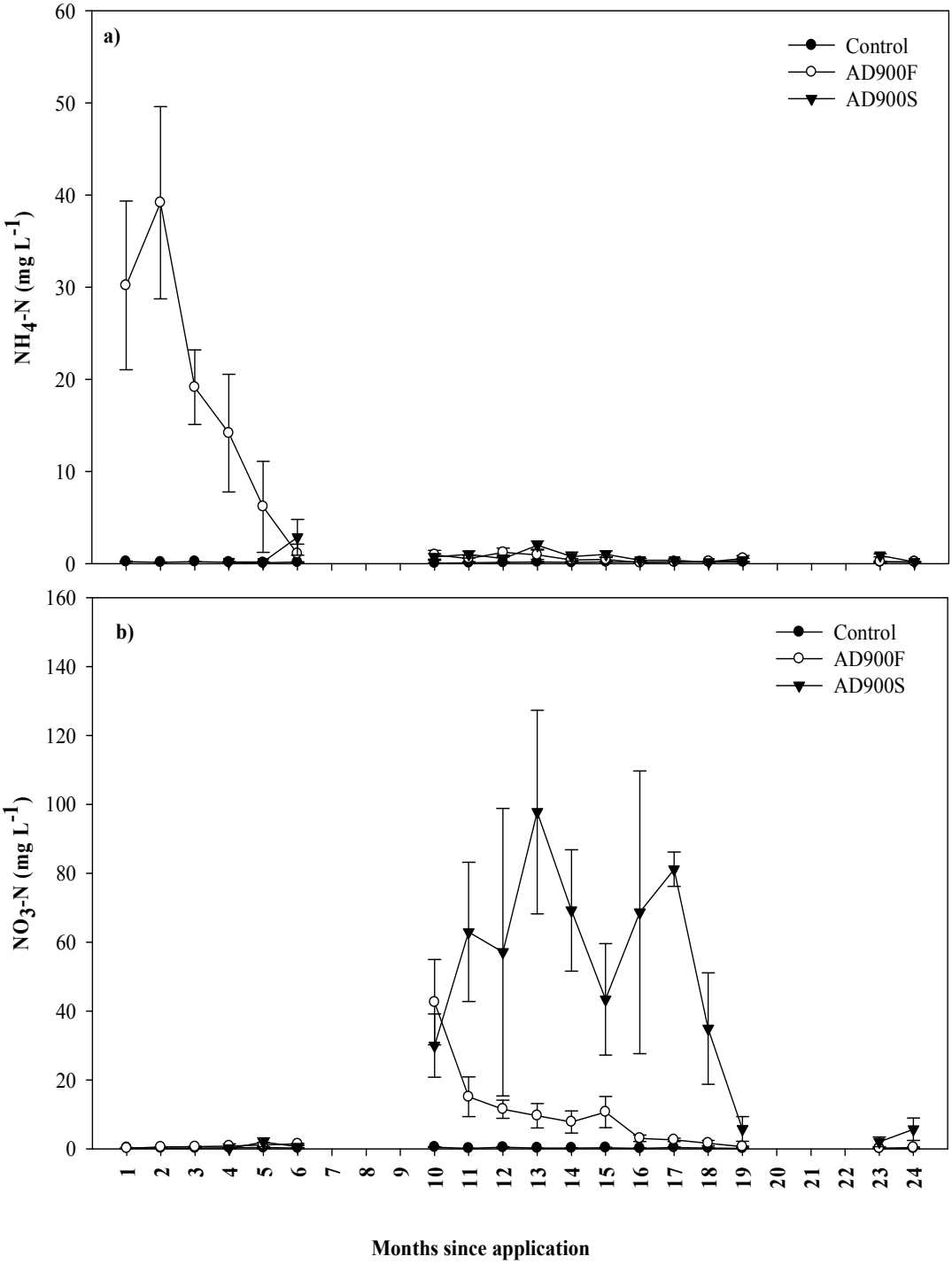
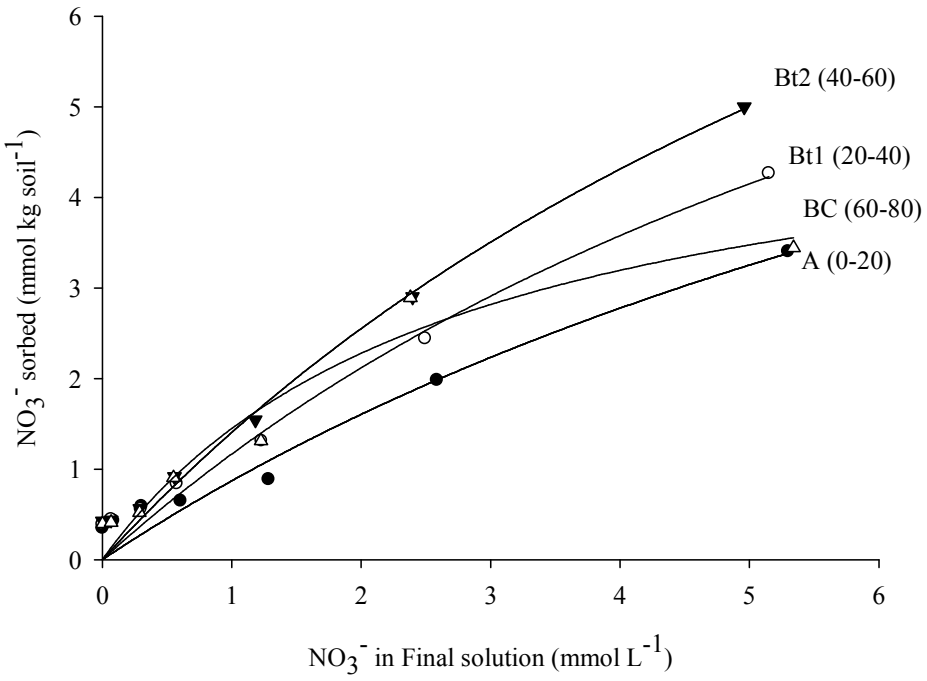


Figure 4.7. NH_4-N (a), and NO_3-N (b) concentration in soil solution from lysimeters sampled over time in a loblolly pine plantation following surface applied anaerobically digested biosolids during November 2005 (fall), and March 2006 (spring) at the rate of 900 kg PAN ha^{-1}. Scale units are different. Brackets indicate ± SE.

Figure 4.8. Quantity of NO_3-N sorbed per kilogram of dry soil across a range of equilibrium NO_3-N solution concentration at natural soil pH for soil collected from a 17 year old loblolly pine plantation at the depth 0 to 20 cm, 20 to 40 cm, 40 to 60 cm, and 60 to 80 cm.



Chapter 5
Short-term Effects of Biosolids Application on Nitrogen and Carbon pools in a Loblolly Pine (*Pinus taeda* L.) Plantation

Abstract

Application of biosolids as an alternative source of N is becoming a common silviculture practices on pine forest. However, little is known about how biosolids type, application rate, and timing affect forest floor and soil N and C pools in loblolly pine plantations. The objectives of this study were to determine changes in C, N, Ca, and pH in the forest floor and the mineral soil after surface application of different types, rates, and season of application of biosolids. The study was established in a 17-year-old loblolly pine plantation in Amelia County, VA. Anaerobically digested (AD225), lime stabilized (LS225) and pelletized (Pellet225) biosolids and a conventional inorganic urea plus diammonium phosphate fertilizer (U+DAP225) were applied at a rate of 225 kg ha^{-1} Plant Available Nitrogen (PAN) between March 5th and 10th, 2006. Anaerobically digested biosolids were also surface applied at the rates of 900 kg PAN ha^{-1} and 1800 kg PAN ha^{-1} (AD900 and AD1800). Anaerobically digested biosolids at the rate of 900 kg PAN ha^{-1} were also applied on November 5th, 2005 (AD900F).

Biosolids application at the 225 kg PAN ha^{-1} rate had little effect on the average total N and C in the forest floor and mineral soil. The average total N in the forest floor in the AD225 treatment was 1005 kg ha^{-1}, 935 kg ha^{-1} in the Pellet225, 852 kg ha^{-1} in the LS225, 398 kg ha^{-1} in the U+DAP225, and 331 kg ha^{-1} in the control. Forest floor total C was greater than the control in the AD225 and the LS225 treatments. The average total C in the forest floor was 20900 kg ha^{-1} in the AD225, 21995 kg ha^{-1} in the LS225, 17864 kg ha^{-1} in the Pellet225, 14420 kg ha^{-1} in the U+DAP225, and 12050 kg ha^{-1} in the control. The application of biosolids significantly reduced C:N in the forest floor and the surface mineral soil in comparison to the control. Total Ca and pH

significantly increased in the forest floor in the AD225 and LS225 treatments in comparison to the other treatments. There was no significant biosolids type effect on extractable Ca in the mineral soil. Soil pH significantly changed over time after application of biosolids.

Higher application rates of biosolids significantly increased forest floor N and C accumulation. The average total N in the forest floor was 1005 kg ha^{-1}, 2075 kg ha^{-1}, and 3199 kg ha^{-1} in the AD225, AD900, and AD1800 treatments, respectively. The average forest floor total C in the AD1800 treatment was 33051 kg ha^{-1}. The AD900 and the AD225 had 28672 kg ha^{-1}, and 20900 kg ha^{-1}, respectively. Total N in the AD1800 was greater than the AD225 and AD900 through all the soil profile. Season of application of biosolids did not significantly affect the forest floor. Soil total C and extractable Ca were greater in the AD900S than in the AD900F. Biosolids have the potential to be use as a source of N to improve tree growth. However, when biosolids were surface applied at the permitted rate of 225 kg PAN ha^{-1}, low impact on soil total N and C were detected. Increasing application rates increased N movement through the soil profile, with no effect on soil carbon.

5.1. Introduction

Land application of biosolids to enhance crops and forest productivity has increased considerably during the last decade. Biosolids application is an effective method to reuse nutrients removed from wastewater treatment plants and can replace commercial fertilizers (Beech et al. 2007). Biosolids are commonly used in agriculture as a source of nitrogen (N), phosphorus (P), and lime (Evanylo 1999a), to restore forest productivity (Henry et al. 1994), and disturbed mine land (Brown and Henry 2000). Consequently, the use of biosolids in agriculture

is a common practice. In 2004, about 224000 dry metric tons of biosolids were land applied in Virginia (Beech et al., 2007).

Several studies identified that the potential for nutrient release from biosolids is determined largely by biosolids chemical composition, pH, initial moisture content and temperature, physical properties, and microbial activity (Cogger et al. 2004; Eldridge et al. 2008; Evanylo 2003; Gilmour et al. 2003; Smith and Durham 2000). The different processes used to treat wastewater sludge such as anaerobic or aerobic digestion, lime stabilization, heat treatment, and composting (Evanylo 1999b), affect the final composition and quality of the biosolids produced (Basta 1995).

Biosolids are typically compose of 40 to 70 % of organic matter (Hartenstein 1981). Typical dried biosolids contain 2–5 % of total N (Evanylo 1999b). Total N in digested liquid material can be close to 10 % (Wang et al. 2004). Biosolids also have a variable amount of other nutrients like P (1-7 %), calcium (Ca) (1-20 %), magnesium (Mg) (0.3-2 %) (Singh and Agrawal 2007; Sommers 1977), and micronutrients (Fiskell et al. 1990; Sommers 1977). Biosolids pH values range from 5.5 to 12 (Maguire et al. 2001; Schroder et al. 2008).

Biosolids have been used as a good alternative to conventional forest fertilization to enhance forest productivity (Prescott and Blevins 2005). Several studies reported the beneficial effect of biosolids on N availability and tree growth (Berry 1987; Egiarte et al. 2005; Hallett et al. 1999; Kelty et al. 2004; Wang et al. 2006). Similar to the results following conventional forest fertilization, surface application of biosolids may increase forest floor total N, and inorganic N availability without affecting mineral soil total N (Hart et al. 1988; Kelty et al. 2004). Biosolids applied to oak forests in northern Michigan enhanced nutrient content in the forest floor, but no significant increases were detected in the mineral soil (Nguyen et al. 1986). No effect on soil

total N have been observed 18-year after conventional fertilization of loblolly pine forest (Johnson et al. 2003).

Applications of biosolids have been shown to increase soil carbon (C) when they are applied to agriculture, forests or reclaimed mine land systems due to the high C content in biosolids (Brown and Henry 2000; Lal 2003). Land application of biosolids have the potential to enhance C sequestration (Brown 2004). Long-term changes in surface soil C were observed after the incorporation of 500 Mg ha^{-1} of biosolids into a coarse texture soil (Harrison et al. 1994a). In contrast, no changes in soil C were observed following surface application of 90 Mg ha^{-1} of biosolids to a grassland system (Moffet et al. 2005). Carbon accumulation in loblolly pine forest is mostly concentrated in the forest biomass, and the forest floor, with minimal accumulation in the mineral soil due to the high C turnover (Richter et al. 1995). Soil C fixation depends on soil texture, mineralogy and the micro-conditions for microbial respiration (Harrison et al. 1995). Loblolly pine growing in a clayey soil fixed larger amount of C in the soil compared with a plantation on a sandy soil (Leggett and Kelting 2006). The same authors reported no effect of forest fertilization on soil C content 11 year following application of 250 kg ha^{-1} diammonium phosphate (DAP) fertilizer.

In the previous two chapters we found significant effect of biosolids on N availability, N mineralization and potential leaching of NO_3-N. This suggests that it could be possible to improve forest soil nutrients and C accumulation following application of biosolids in a loblolly pine plantation (Harrison et al. 1994a). In this chapter we considered changes in N and C pools after surface application of biosolids, and the impact on N and C accumulation of the forest floor and mineral soil. We also followed changes in pH and Ca content to increase the understanding of the effect of lime addition through different type of biosolids. Therefore, the objective of this

study was to determine changes in nutrient pools after surface application of biosolids to a 17-year-old loblolly pine plantation. The specific objectives of this study were to determine the difference in C, N, Ca, and pH in the forest floor and the mineral soil after surface application of different type of biosolids, different rates, and season of application on nutrient content in the soil profile.

5.2. Materials and Methods

5.2.1. Study Area

The study was established in the summer of 2005 in Amelia County northeast of Blackstone, VA (37° 13' N, 77° 48' W). The site is located in the Piedmont physiographic province and supported a 17-year-old loblolly pine plantation. The stand was thinned in 2005 using a combination of fifth-row removal and low thinning between the removed rows. The mean annual temperature is 14 °C, with a mean of 4 °C in January and 25 °C in July. The mean annual precipitation is 113 cm with July and August being the wettest. The mean snowfall is 30 cm concentrated between December and March (15-year period). The local climatologic recent records were obtained from the closest weather station in Blackstone, VA which was approximately 40 km from the study site. During the month of the spring application (March 2006), precipitation was only 0.68 cm, which was below the historical average 8.1 cm for the area (Fig 5.1). Conditions were near normal throughout most of the spring and summer of the same year, while rainfall from August to November 2006 was greater than normal.

The soil at the site is the Appling series (Fine, kaolinitic, thermic Typic Kanhapludults). Slopes range from 2% to 5%. The thin Ap layer shows evidence of surface soil erosion caused by past agriculture practice, and in some areas is mixed with the argillic Bt horizon. Soil samples

collected at four different depths, from 0 to 20 cm, 20 to 40 cm, 40 to 60 cm, 60 to 80 cm prior to treatments were analyzed for total C and N by combustion using a CNS analyzer (Elementar America, Inc, Laurel, NJ), and Mehlich-1 extractable P, K, Ca, Mg using a ICAP-AES following the Virginia Cooperative Extension Methodology (Donohue and Heckendorn 1994; Mehlich 1953). Soil pH was determined in a 1:1 soil:water ratio. Soil cores were collected with a bulk density hammer and subsequently oven dried and weigh to measure bulk density (Table 5.1).

5.2.2. Experimental Design and Treatments

The experimental design was a randomized complete block design with four blocks and eight treatments comparing biosolids type, granular fertilizer, rates, and season of application. Thirty-six plots of 0.45 ha (150 x 30 m) separated by a 30 m of untreated buffer area were established in September 2005. Three different types of biosolids were evaluated; lime stabilized, anaerobically digested, and pelletized biosolids. The anaerobically digested material was obtained from the Alexandria, VA and Back River, MD wastewater treatment facilities, for the fall and spring application, respectively. The lime stabilized biosolids were obtained from the Blue Plains wastewater treatment facility in Washington, DC. The pelletized biosolids were obtained from the Synagro pelletized facility in Back River, MD. The granular fertilizer treatments were a mix of conventional urea-N (46-0-0) and DAP (18-46-0) obtained from Southern States Cooperative, Inc., (Christiansburg, VA). Physical and chemical properties of the biosolids are presented in Table 5.2.

Anaerobically digested, lime stabilized, pelletized biosolids and a conventional inorganic fertilizer were applied at a rate of 225 kg ha^{-1} Plant Available N (PAN). In order to understand the consequences of application rates greater than the recommended rate of 225 kg ha^{-1} PAN, anaerobically digested biosolids were added at 900 and 1800 kg ha^{-1} PAN. These treatments were

applied between March 5th and 10th, 2006. To test the impact of season of application timing (fall vs. spring), the anaerobically digested biosolids were also applied at 900 kg ha^{-1} PAN on November 5th, 2005. Treatments descriptions and final application rates are listed in Table 5.3.

Biosolids application rates were estimated based on the average N concentration of samples analyzed during the six previous months before the beginning of the study, and corroborated with field samples. The treatments rates were based on PAN approach, determined according to recommendations established for VA when biosolids are surface applied (Evanylo 1999a).

$$\mathbf{PAN = NO_3\text{-}N + \mathit{Kvol}\ (NH_4\text{-}N) + \mathit{Kmin}\ (Org\text{-}N)}$$

Where:

PAN = Kg of Plant Available N dry^{-1} Mg biosolids

NO_3-N = Kg nitrate-N dry^{-1} Mg biosolids

Kvol = volatilization factor, or plant-available fraction of NH_4-N (lime stabilized = *0.25*, anaerobically digested = *0.5*)

NH_4-N =Kg ammonium-N dry^{-1} Mg biosolids

Kmin = mineralization factor, or plant-available fraction of Org-N (lime stabilized = *0.3*, and anaerobically digested = *0.2*)

Org-N = Kg organic-N dry^{-1} Mg biosolids (estimated by organic N = total Kjeldahl-N - NH_4-N)

The biosolids were transported from each wastewater treatment plant, piled at the site and applied during the same day. Biosolids were surface applied using a skidder with a side discharge spreader that went through the previous thinned corridors in the stand. The biosolids were not tilled into the soil. Four plastic collection trays (60 x 20 cm) were installed in each plot

prior to application. The biosolids collected in each treatment were weighed, and moisture content was determined after the application. Samples were collected from the trays in the field and transported to A&L Eastern Agricultural Laboratories (Richmond, VA) for routine biosolids test analysis for total and volatile solids (SM2540G), nitrate-N (SM4500- NO_3-F) (APHA 1998), pH according to SW 846-9045C (USEPA 2002), and calcium carbonate equivalent (CCE) using AOAC 955.01 (Kane 2000). Total Kjeldahl nitrogen (USEPA 351.3) and ammonium-N (USEPA 350.2) (USEPA 1983). Phosphorus (P), potassium (K), sulfur (S), calcium (Ca), magnesium (Mg), sodium (Na), iron (Fe), manganese (Mn), cadmium (Cd), copper (Cu), lead (Pb), molybdenum (Mo), nickel (Ni), and zinc (Zn) were measured according to SW846-6010B (USEPA 2002) (Table 5.2).

Conventional granular fertilizer treatments were broadcast at the same time using a backpack spreader so the fertilizer evenly covered the forest floor. The competing herbaceous understory vegetation was removed chemically using a foliar application of 5% Round-up Pro ™ (Monsanto Co, St Louis, MO) applied with a backpack sprayer in all plots during the summer of 2005 and 2006.

5.2.3. Forest Floor and Soil Samples

Prior to biosolids application forest floor and soil were sampled (Table 5.2). In February of 2007 and 2008 soils were sample at the depth of 0 to 20 cm (Ap), 20 to 40 cm (Bt), 40 to 60 cm (Bt), and 60 to 80 cm (BC) using a soil auger. Three samples were collected at each plot and depth. The three samples from each depth were composited in each plot. All soil samples were air dried and passed through a 2-mm screen prior to analysis. A bulk density soil sampler was used to obtain 5 cm x 10 cm undisturbed cores at each depth. Bulk density samples were dried at

105 C and the dry mass of soil was used to determine bulk density. The forest floor was collected at the time of soil sampling within a 0.25-m^2 sample area, sieved through 4mm sieve, dried in a forced draft oven at 75 °C for 10 days, weighed, and ground in a Wiley mill to pass through a 20-mesh screen.

Total forest floor and soil N and C were determined by dry combustion using an Elementar CNS analyzer (Elementar, Hanau, Germany). Extracted soil Ca was determined following extractions using the Mehlich I method (Mehlich 1984) .Total Ca in forest floor was determined following ashing at 500 °C and dissolving the ash in 0.1 N HCl (Jones and Steyn 1973). The resulting solutions for the forest floor and soil samples were analyzed using an Inductively Coupled Plasma analyzer (Varian Vista MPX, Varian Instruments, Cary, NC). Forest floor and soil pH was measured in a 1:1 (vol) H_2O solution. Total C, N, and Ca in the forest floor were expressed as kg ha^{-1} based on the mass of forest floor collected in the sampling area. Total C and N, and extractable Ca were express as kg ha^{-1} based on depth of each horizon and bulk density in each mineral soil horizon.

5.2.4. Statistical Analysis

Forest floor and soil were analyzed separately. Data were analyzed using analysis of variance accounting for treatment, soil depth, and time as factors using the Proc Mixed Procedure in SAS 9.1 (SAS, Cary, NC). Repeated measurements were conducted to account for the time effect of the 2007 and 2008 samples (Littell et al. 2006). Test for normality, linearity, and constant variance of the residuals were performed. Logaritmic transformations were necessary in forest floor Ca analyses to ensure the validity of the assumptions. Results are presented in untransformed values. Fisher LSD multiple comparison test was used to determine

significant difference among means at $\alpha = 0.05$ when the interaction and/or main effect were significant in the ANOVA ($P<0.05$ in F test). Linear contrasts were performed to compare the main effect of application treatments throughout the study.

5.3. Results

Application of biosolids significantly affected forest floor total N, total C, C:N, total Ca, and pH in the forest floor. There was a significant time effect on total N and Ca accumulated in the forest floor, while there was a significant treatment time interaction for forest floor pH (Table 5.4).

Biosolids also significantly affected total N, total C, C:N, and pH in the mineral soil There was a significant treatment time interaction for the soil total C, extractable Ca, and pH (Table 5.4). There was a significant effect of soil depth in total N, total C, C:N, Ca and soil pH (Table 5.4). Like most of the soils in the Piedmont region, nutrients content decreased by soil depth.

5.3.1. Effect of Biosolids Type on Forest Floor and Soil

5.3.1.1. Total N and C

No significant differences were detected in the forest floor N content when biosolids were added at the 225 kg ha^{-1} PAN rate (Table 5.5). The average forest floor total N in the AD225 treatment was 1005 kg ha^{-1}, 935 kg ha^{-1} in the Pellet225, 852 kg ha^{-1} in the LS225, 398 kg ha^{-1} in the U+DAP225, and 331 kg ha^{-1} in the control. Total N in the forest floor declined over time in the AD225 treatment (Fig 5.2.a).

The average forest floor total C was significantly greater than the control in the LS225 treatment. Our results suggested that the average total C in the AD225 treatment was also greater than the control at the significant level $p < 0.1$ (Table 5.5). The average total C in the forest floor was 20900 kg ha^{-1} in the AD225, 21995 kg ha^{-1} in the LS225, 17864 kg ha^{-1} in the Pellet225, 14420 kg ha^{-1} in the U+DAP225, and 12050 kg ha^{-1} in the control. We observed significant treatment time interaction. In 2007, forest floor total C was greater in the AD225 ($p = 0.0355$), and the LS225 ($p = 0.045$) treatments relative to the control. By the second sample date in 2008, only the total C in the LS225 treatment remained significantly greater than the control in the forest floor ($p = 0.045$) (Fig 5.3.a).

The application of Pellet225 significantly increased the average soil total N in comparison to the control treatment (Table 5.5). There was a significant depth effect, with most of the N content located in the surface 20 cm of soil (Fig 5.2). The biosolids treatments and the control had similar soil total N. The average total N in the soil profile in the Pellet225 was 667 kg ha^{-1}, in the U+DAP225 treatment was 642 kg ha^{-1}, the LS225 was 589 kg ha^{-1}, and in the AD225 treatment was 557 kg ha^{-1}.

The average soil total C was significantly greater than the control in the LS225 treatment (Table 5.5). There was a significant treatment time interaction effect. In 2007, the soil total C in the Pellet225 was lower than the U+DAP225. By 2008, there were no significant differences among any of the treatments (Fig 5.3). The average soil total C was 7745 kg ha^{-1} in the AD225, 8440 kg ha^{-1} in the LS225, 7595 kg ha^{-1} in the Pellet225, 8155 kg ha^{-1} in the U+DAP225, and 7137 kg ha^{-1} in the control treatment. Soil total C decreased with soil depth in all the treatments (Fig 5.3).

The application of biosolids significantly affected the C:N in both the forest floor and the mineral soil (Table 5.4). In the forest floor, the higher N content in the biosolids treatments significantly reduced C:N in comparison to the control and the U+DAP225 treatments (Table 5.5, Fig 5.4). The average C:N was 37 in the control treatment, while it dropped to 20.3 in the Pellet225, 26.3 in the LS225, 21.6 in the AD225, and 36.4 in the U+DAP225. C:N in the forest floor tended to stay relative similar during time (Fig 5.4.a).

The increase in total N in the mineral soil significantly reduced the average C:N in the Pellet225 in comparison to the control, LS225, and AD225 treatments (Table 5.5). The average C:N in the Pellet225 was 12, in the AD225 was 13.5, in the LS225 was 16.1, in the U+DAP225 14.1, and the control had a C:N of 14.9.

5.3.1.2. Total Ca and pH

Biosolids type significantly affected forest floor total Ca and pH. The average total Ca in the LS225 treatment was significantly higher than the control, and all the other treatments. Total Ca accumulated in the forest floor was also greater in the AD225 than in the U+DAP225, and control treatment (Table 5.5, Fig 5.5.a). The average total Ca in the forest floor was 1266 kg ha^{-1} in the LS225 treatment, 311 kg ha^{-1} in the AD225, 270 kg ha^{-1} in the Pellet225, and 87 kg ha^{-1} in the U+DAP225.

The forest floor pH response was directly affected by the contribution of the total Ca (Fig 5.5.a, Fig 5.6.a). The average forest floor pH was 6.3 in the LS225, 5.2 in the AD225, 5.1 in the Pellet225, 4.8 in the U+DAP225, and 4.6 in the control treatment. The average forest floor pH in the LS225 treatment was significantly higher than all the other treatments in both years (p values <0.001) (Table 5.5). The average forest floor pH in the AD225 and the Pellet225 treatment was

significantly higher than the U+DAP225 and the control treatments (Table 5.5). Forest floor pH declined in the 2008 samples in all the biosolids treatments. The pH in the AD225 and Pellet225 treatments was greater than the control and the U+DAP225 only in 2007 (Fig 5.6.a).

In contrast, there was no significant biosolids type effect on extractable Ca in the mineral soil (Table 5.5, Fig 5.5). Soil pH significantly changed over time after application of biosolids. The average soil pH was 5.7 in the AD225, 5.5 in the LS225, 5.5 in the Pellet225, 5.4 in the U+DAP225, and 5.5 in the control treatment. There was a significant treatment time interaction effect of soil pH. In 2007, soil pH in the AD225 was greater than the LS225 ($p = 0.0121$), U+DAP225 ($p = 0.0029$), and the control ($p = 0.045$). In 2008, pH in the LS225, U+DAP225, and control tended to increase, while the pH in the AD225 decreased (Fig 5.6). In the 2008 sample, there were no differences among treatments.

5.3.2. Effect of Biosolids Application Rates

5.3.2.1. Total N and C

The application of higher rates of biosolids significantly increased total N in the forest floor during both years of measurements (Table 5.5, Fig 5.7.a). The average forest floor total N in the AD1800, and AD9000 treatments was significantly higher than AD225. Total N in the AD1800 was significantly greater than in the AD900 (Table 5.5). The average total N was 3199 kg ha^{-1}, 2075 kg ha^{-1}, and 1005 kg ha^{-1} in the AD1800, AD900, and AD225 treatments, respectively. Total N declined by 2008 in the AD1800 treatment, but still remained greater than in the AD900 and the AD225 treatments (Fig 5.7.a).

Similar to the total N content in the forest floor, the total C in the forest floor increased with increasing biosolids application rates and decreased over time (Fig 5.8.a). The average

forest floor total C in the AD1800 treatment was 33051 kg ha^{-1}. The AD900 and the AD225 had 28672 kg ha^{-1}, and 20900 kg ha^{-1} total C, respectively. The average forest floor total C in the AD1800 was significantly higher than the AD225 in both years and no significant differences in total C were detected among the other treatment rates (Table 5.5). Total C significantly declined over time in the AD1800 treatments (Fig 5.8.a).

Soil total N content also increased with application rates. However, only the AD1800 treatment had a significantly higher average total N content than the AD225, and AD900 treatments (Table 5.5). The average total N in the mineral soil in the AD1800 was 839 kg ha^{-1}, in the AD900 was 568 kg ha^{-1}, and in the AD225 was 557 kg ha^{-1}. The AD1800 had significantly higher level of total N than the other two rates in all the soil horizons (p values < 0.0261) (Fig 5.7.b, 5.7.c, and 5.7.d).

The average total C in the mineral soil did not change with increasing application rates (Table 5.5) (Fig 5.8). Soil total C tended to increase over time and decrease by sample depth (Fig 5.8).

The application of anaerobically digested biosolids significantly increased total N, and altered forest floor and mineral soil C:N values (Table 5.5). Increasing rates significantly reduced the forest floor C:N to 11, 13, and 21 in the AD1800, AD900, and AD225 treatments, respectively. The average C:N in the AD225 was significantly higher than the AD1800 and the AD900 treatments. No differences were detected between the AD900 and AD1800 (Table 5.5 Fig 5.9.a). In the mineral soil, the AD1800 had an average C:N of 9.4 and it was significantly lower than the AD225 (13.5), and the AD900 (16.9) treatments (Table 5.5).

5.3.2.2. Ca and pH

Total Ca in the forest floor increased with increasing biosolids application rates (Fig 5.8.b). The average total Ca accumulated in the forest floor in the AD1800, and the AD900 treatments was significantly greater than in the AD225 treatment (Table 5.5). The average total Ca in the forest floor was 311 kg ha^{-1} in the AD225, 625 kg ha^{-1} in the AD900, and 1044 kg ha^{-1} in the AD1800 treatment. Forest floor pH was similar during the first year among all the treatments. The average forest floor pH was greater in the AD900 compared to the three application rates had an average forest floor pH of 5.2, 5.3, and 5.4 in the AD225, AD900, and AD1800 treatments, respectively. Forest floor pH values significantly declined in the AD225 treatment in 2008, and it was significantly lower than the AD1800 treatment ($p = 0.032$) (Fig 5.11.a).

There was no significant effect of application rate on the average extractable Ca in the mineral soil (Table 5.5). However there was a significant treatment time interaction effect on soil extractable Ca. In 2007, total extractable Ca was greater in the AD225 compared to the AD1800 ($p = 0.0006$) and the AD900 ($p = 0.0051$) treatments. Total extractable Ca significantly declined from 2007 to 2008 in the AD225 treatment. In contrast the extractable Ca increased over time in the AD1800 treatment. In 2008, the extractable Ca was significantly greater than the AD225 ($p = 0.048$) and the AD900 ($p = 0.0491$) treatments (Fig 5.10).

Increasing biosolids application rates significantly affected the average soil pH (Table 5.5). The average soil pH was 5.2, 5.4, and 5.7 in the AD1800, AD900, and AD225, respectively. There was a significant treatment time interaction. The AD225 was significantly greater than the AD1800 and AD900 in both sampling dates (Fig 5.11.b, 5.11.c, 5.11.d). Soil pH in the AD900S significantly dropped from 2007 to 2008.

5.3.3. Effect of Season of Biosolids Application

Season of application of biosolids did not significantly affect total N, total C, C:N , total Ca and pH measured in the forest floor (Tables 5.5). In contrast, season of application significantly affected soil total C, C:N, and extractable Ca (Table 5.5, Fig 5.13, 5.14, and 5.15) There was no effect of season of application on soil total N and soil pH (Fig 5.12, and 5.16). The average soil total C in the AD900S was 8880 kg ha^{-1}, and it was greater than the AD900F which had 7449 kg ha-^{1}. The C:N in the AD900S was 16.9 and 13.5 in the AD900F treatment. The average extractable Ca in the AD900F was 918 kg ha^{-1} and 778 kg ha^{-1} measured in the AD900S treatment.

5.4. Discussion

5.4.1. Effect of Biosolids Type on Total N and C pools

We observed that application of different type of biosolids had little effect on the total N in the forest floor 23 months following application (Table 5.5 Fig 5.2). Several studies identified large accumulation of nutrients in the forest floor after surface application of biosolids (Bramryd 2002; Harrison et al. 1995; Hart et al. 1988; Kelty et al. 2004; McLaren et al. 2007). The high variability observed on the N, C and Ca content in the forest floor is partly explained by the uneven distribution of biosolids when they were surface applied and the variable total biosolids dry mass applied on each treatment (Table 5.3).

Nitrogen losses from surface application of biosolids can be interpreted by the sum of tree uptake, volatilization, leaching, denitrification, and immobilization after N mineralization (Hallett et al. 1999; Magesan et al. 1998; Medalie et al. 1994; Robinson and Polglase 2000;

Wang et al. 2004). When biosolids were surface applied to radiata pine (*Monterey pine*) forest, the major cause of N losses was volatilization (Robinson and Roper 2003). Anaerobically digested biosolids were shown to lose 60% of available N through volatilization during the five-day period following application (Beauchamp et al. 1978), and between 71 to 81% within a 3 weeks period when they were surface applied to forest (Robinson et al. 2002).

In this study, the organic-N content in biosolids was 97%, 95%, and 77% of the total N applied in the LS225, Pellet225, and AD225 treatments, respectively. The observed initial organic-N content in biosolids and the declined through time in total N in the forest floor indicate that apparently N addition depended mostly on mineralization. Several studies shown that N mineralization of the labile N in biosolids is more active during the first year and the remaining N bond to the recalcitrant component in the forest floor (Brockway et al. 1986; Haith et al. 1992; Kaufman and Haith 1986). The remaining N in biosolids could be estimated by subtracting the forest floor total N in the control treatment from the AD225 and the LS225 treatments. The remaining N was 58% and 50%, in the AD225 and LS225, respectively. Estimates from different type of forests shown that 45% of the total N in biosolids applied to hardwood forests remained in the surface soil at the end of two years (Nguyen et al. 1986), and 65% in Douglas fir forest floor two years following application of biosolids (Edmonds and Mayer 1981).

We observed little effect on soil total N after surface application of biosolids. This agrees with other studies were biosolids were surface applied at similar rates (Hallett et al. 1999; Robinson et al. 2002). Egiarte et al (2005) found no response on total C and N in the surface soil one year after two consecutive applications of 2.4, 17, and 60 Mg ha^{-1} of anaerobically digested biosolids in a radiata pine forest. In contrast, long term effect on total N have been observed when biosolids were disked at the time of application (Harrison et al. 1995). In this study, our

targeted application rate was based on estimates of soil N availability as a fertilizer for tree growth. The proportion of inorganic N added through fertilization is relatively small in relation to the total N in the soil (Vitousek and Matson 1985).

Only the Pellet225 treatment significantly increased soil total N in the mineral soil. Rapid N mineralization have been reported by Tarrason et al (2008) when they compared thermally dried biosolids with anaerobically digested biosolids, and it is explained by the high proportion of labile organic N in pelletized and thermally dried biosolids (Smith and Durham 2002; Smith et al. 1998). It could be possible that N release from pellets got incorporated faster in the surface soil than the other biosolids treatments. Eldridge et al (2008) determined that about 50% of N mineralization occurred within two months after surface application of pelletized biosolids. Kelty et al. (2004) reported that 26% of the organic N applied with pelletized biosolids was rapidly mineralized following surface application in red pine forest.

Consequently with the forest floor N content results, total C in the forest floor declined over time in the biosolids treatments. The main source of carbon losses from surface applied biosolids occurred as a consequence of oxidation and the rapid C turnover through mineralization. Robinson et al. (2002) reported 52 to 67% of carbon losses in 2 years following surface application of biosolids to radiata pine forest. No changes in soil carbon in a loblolly pine forest have been observed when conventional fertilizers were applied to loblolly pine forest and measured sixteen months (Carter et al. 2002) or eleven years (Leggett and Kelting 2006) following fertilization. The low C incorporation through the soil profile indicated no potential for soil C sequestration when biosolids are surface applied as an alternative source of N. Fertilization increased carbon sequestration in loblolly pine plantation as a consequence of

biomass growth but not due to gains in soil carbon content (Leggett and Kelting 2006; Luxmoore et al. 1999).

The C:N ratios are commonly used to describe organic matter decomposition in biosolids (Gilmour and Skinner 1999) and N cycling in forest ecosystems (Ollinger et al. 2002). The C:N of the forest floor was close to 37 in the control treatment. Measurements 11 months after biosolids application showed that C:N declined to values between 19.2 and 23.5 in the biosolids treatments. McLaren et al. (2007) reported similar forest floor C:N values when biosolids were surface applied in a 25-year-old radiata pine plantation. Our C:N results suggested slow mineralization rate in the biosolids treatments (Henry et al. 1999). At the time of sampling most of the N mineralization of the active pool probably already occurred (Gilmour and Skinner 1999), as biosolids have a low initial C:N. Parnaudeau et al. (2004) established that C:N ratio of 17 different types of biosolids ranged between 5 and 19. In the second year of measurements, the C:N in biosolids remained stable in the AD225, and the Pellet225 treatments, which suggest that mineralization is still occurring at a low rate (Henry, 1999).

Total C and C:N is not always an indication of how easily biodegradable biosolids may be (Cabrera et al. 2005). Several studies indicated that decomposition dynamics in biosolids depends on the type of organic compounds (Parnaudeau et al. 2006; Smith et al. 2008). . Different biosolids type have a variable concentration of labile and recalcitrant carbon that affected microbial decomposition (Hartenstein 1981). In general, as lignin content in biosolids increased, decomposition decreased (Hattori and Mukai 1986) Rates of net N mineralization in biosolids were best predicted by a model incorporating the initial organic N concentration and the proportion of phenolic C (Rowell et al. 2001). Anaerobically digested biosolids are more stable than aerobically digested biosolids. Field and laboratory incubations had found that

anaerobically digested biosolids have more stable organic structures that decreased mineralization (Donovan and Logan 1983; Wang et al. 2003).

Our results have also demonstrated that Ca content and pH in the forest floor increases following surface application of biosolids to forest as shown in the high Ca content and the significant increase in pH in the forest floor (Fig 5.5 and 5.6). However, there was a limited effect on soil extractable Ca. The initial response in forest floor pH was expected since biosolids applied in the AD225 and LS225 treatments had a pH of 8.1 and 12.3, respectively. Both biosolids type shown calcium carbonate equivalence (CCE) and are use as a source of lime to increase soil pH (Table 5.2). The effect of lime addition to biosolids directly increased Ca content in relation with unlimed biosolids (Maguire et al. 2001)

The forest floor interaction effect in the total Ca and pH was also observed when alkaline biosolids were applied to Sitka spruce (*Picea sitchensis*) (Luo and Christie 2001) and radiata pine (McLaren et al. 2007) and it is attribute to the slow Ca release from biosolids and the cation exchange capacity of the forest floor. The lack of response in soil extractable Ca could be explained by the sampling depth. Luo and Christie (2001) found significant changes in soil Ca concentration mostly in the top 5 cm of the mineral soil.

Soil mineralogy, previous land use, and method of incorporation affected soil pH response to lime addition (Mullins et al. 2005). Frank and Stuanes (2003) observed significant effect on soil pH of lime addition in Scots pine (*pinus sylvestris* L) forest growing in an acid sandy soil. The low soil Ca and pH response in this study was probably due to the combine effect of forest floor, the relatively high natural soil pH, and the slow Ca incorporation when lime sources material like biosolids are surface applied without disking (Mullins et al. 2005). Forest floor pH declined over time, probably as a consequence of the Ca leaching into the soil (McLaren et al.

2003), and the acidification effect of nitrification of the NH_4-N release from the N mineralization occurring in biosolids (Pocknee and Sumner 1997).

5.4.2. Effect of Biosolids Application Rate

Total N in the forest floor and soil was much higher in the high application rates in comparison to the biosolids rate applied in the AD225 treatment. This is not surprising since the total N accumulated in the forest floor and mineral soil was directly proportionate to the total N applied with the biosolids. The significantly increased in total N through the soil profile in the AD1800 treatment compared to the other two application rates suggest that significant amount of N is leaching through the soil profile. This is also support by the low C:N in the forest floor and mineral soil that suggest a rapid mineralization and N movement through the soil profile. Nitrogen leaching and accumulation through the soil profile have been shown when high rates of biosolids are applied to forest (Brockway and Urie 1983; Egiarte et al. 2005; Hallett et al. 1999; McLaren et al. 2005; Robinson et al. 2002). Brockway and Urie (1983) reported NO_3-N leaching above water quality standards 2 years after application of anaerobically digested biosolids in a pine stand receiving over 19.3 dry Mg ha^{-1} or and aspen sprout stands receiving rates over 23 dry Mg ha^{-1}.

We found no C accumulation in the forest floor as a consequence of high application rates. Similar results were observed when 34 and 90 Mg ha^{-1} of biosolids were surface applied in grassland and shrubland growing in aridic conditions (Moffet et al. 2005). For the same site, Rostagno and Sossebee (2001) found no effect on soil organic matter deeper than three cm when 90 Mg ha^{-1} of biosolids were surface applied. In contrast, large long-term accumulations of soil C in the surface soil have been observed when high application rates of biosolids were

incorporated into the forest soil (Harrison et al. 1994a) or applied to heavily disturbed sites (Malik and Scullion 1998). Two things explain our low impact on soil C content. First, the limited C incorporation into the soil and the high C losses when biosolids are surface applied (Robinson et al. 2002), and the large variability in soil C measurements, which have been observed in loblolly pine stands growing in disturb land converted from agriculture (Van Lear and Kapeluck 1995).

We showed that soil extractable Ca significantly increase over time in the AD1800 treatment compared to the AD900 and the AD225 rates through the whole soil profile (Fig 5.15). Calcium movements through soil profile have been associated with high application rates of biosolids. Riekerk (1978) reported 15% of Ca losses from biosolids, and high concentration of $CaNO_3$ in ground water one year after surface application of 247 metric ton ha^{-1} of biosolids in Douglas fir forest. We also found that soil pH declined over time after high application rates (Fig 5.16). Application rates that exceeds plant N uptake have been shown to increase soil NO_3-N concentration, soil acidification, and cation leaching (Harrison et al. 1994b). These authors also reported that increasing application rates reduce tree growth and site quality.

5.4.3. Effect of Season of Biosolids Application

We found that application of biosolids have no significant effect on forest floor total N, C, C:N, Ca and pH. These results were not surprising since the application rates and biosolids type were the same, and the application dates were only 4 months apart. By the time of sampling a large portion of the labile pool of nutrients was probably already mineralized in both treatments, and only a low fraction is still mineralizing (Henry et al. 1999). We found that soil C and Ca was higher in the spring application treatments in comparison to the fall application. These

differences are more likely produce by the high variability found in soil carbon content on managed forest (Johnson and Curtis 2000) since there was no forest floor response attributed to treatments application.

5.5. Conclusions

We found that surface application of biosolids had relatively little impact on total N and C in the forest floor or the mineral soil one or two years following treatment application in a loblolly pine forest. When biosolids were applied at the permitted rate of 225 kg PAN ha^{-1}, we observed evidence of C accumulation in the forest floor but little impact in the surface soil. Specific biosolids properties affected total Ca and pH in the forest floor. Because of the addition of Ca in the wastewater treatment process in biosolids used in the LS225 and AD225 treatments, large amounts of Ca accumulated in the forest floor. Despite the liming potential recognized in biosolids, soil pH slightly increased in the mineral soil, and by the second measurements soil pH returned to control levels.

We found evidence that conditions for N mineralization from biosolids persisted two years following application of biosolids. The declined in C:N in the forest floor in all the biosolids treatments suggested that surface application of biosolids have the potential to release N for more than one growing season. However, several studies indicated that decomposition in biosolids depends on the relation between the labile and recalcitrant organic N forms. Nitrogen release from N mineralized in biosolids is not always available for tree uptake.

When biosolids were applied at increasing rates, we observed large accumulation of N and C in the forest floor but little effect on soil total C. Our results suggested N accumulation through the soil profile with the rate applied in the AD1800 treatments. The significant increased in N

content and extractable Ca through the soil profile, and the declined in soil pH showed that there are strong evidences of high nitrification activity and NO_3-N and cation leaching. Biosolids applications at high rates provided no gain in soil carbon sequestration and increase the potential for groundwater pollution and nutrients leaching.

Biosolids have the potential to be use as a source of nutrient in loblolly pine forest. However, in order to incorporate biosolids application as a source of N and increase the potential for C sequestration we need to increase the understanding of how site-specific characteristics, biosolids type, and method of biosolids incorporation improve forest site productivity without decreasing environmental quality.

5.6. REFERENCES

APHA. 1998. Standard methods for examination of water and wastewater. Am Water Works Assoc., and Water Environment Federation, Washington, DC.

Basta, N.T. 1995. Land application of biosolids : a review of research concerning benefits, environmental impacts, and regulations of applying treated sewage sludge. Oklahoma Agricultural Experiment Station : Center for Agriculture and the Environment, Division of Agricultural Sciences and Natural Resources, Oklahoma State University, [Stillwater]. xii, 59 p. p.

Beauchamp, E.G., G.E. Kidd, and G. Thurtell. 1978. Ammonia volatilization from sewage sludge applied in field. Journal of Environmental Quality 7(1):141-146.

Beech, N., K. Crawford, N. Goldstein, G. Kester, M. Lono-Batura, and E. Dzieyk. 2007. A national biosolids regulation, quality, end use and disposal survey: Final report. North East Biosolids and Residuals Association (NEBRA). 30.

Berry, C.R. 1987. Use of municipal sewage sludge for improvement of forest sites in the southeast. United Stated Department of Agriculture, Forest Service. 33 pp.

Bramryd, T. 2002. Impact of sewage sludge application on the long-term nutrient balance in acid soils of Scots pine (Pinus sylvestris, L.) forests. Water Air and Soil Pollution 140(1-4):381-399.

Brockway, D.G., and D.H. Urie. 1983. Determining Sludge Fertilization Rates for Forests from Nitrate-N in Leachate and Groundwater. Journal of Environmental Quality 12(4):487-492.

Brockway, D.G., D.H. Urie, P.V. Nguyen, and J.B. Hart. 1986. Wastewater and sludge nutrient utilization in forest ecosystems. P. 221-245 in The forest alternative for tWastewater and sludge nutreatment and utilization of municipal and industrial wastes, D.W., C., C. Henry, and N. W.L. (eds.). University of Washington Press, Seattle.WA.

Brown, D. 2004. Building carbon credits with biosolids recycling. BioCycle: Journal of Composting & Organic Recycling. September:25-29.

Brown, S., and C. Henry. 2000. Using biosolids at mine reclamation sites. BioCycle: Journal of Composting & Organic Recycling 41(2):18-19.

Cabrera, M.L., D.E. Kissel, and M.F. Vigil. 2005. Nitrogen mineralization from organic residues: Research opportunities. Conference on Sustainable Land Application:75-79.

Carter, M.C., T.J. Dean, M. Zhuo, M.G. Messina, and Z. Wang. 2002. Short-term changes in soil C, N, and biota following harvesting and regeneration of loblolly pine (Pinus taeda L.). Forest Ecology and Management 164:67-88.

Cogger, C.G., A.I. Bary, D.M. Sullivan, and E.A. Myhre. 2004. Biosolids processing effects on first- and second-year available nitrogen. Soil Science Society of America Journal 68(1):162-167.

Donohue, S.J., and S.E. Heckendorn. 1994. Soil test recommendations for Virginia. Virginia Tech. Blacksburg, VA.

Donovan, W.C., and T.J. Logan. 1983. Factors affecting ammonia volatilization from sewage-sludge applied to soil in a laboratory study. Journal of Environmental Quality 12(4):584-590.

Edmonds, R.L., and K.P. Mayer. 1981. Survival of sludge-associated pathogens and their movement into groundwater. P. 79-86 in Municipal sludge applicarion to Pacific Northwest forest land, Bledsoe, C.S. (ed.). Institute of Forest Resource, University of Washington, Seattle, WA.

Egiarte, G., M.C. Arbestain, A. Alonsoa, E. Ruiz-Romera, and M. Pinto. 2005. Effect of repeated applications of sewage sludge on the fate of N in soils under Monterey pine stands. Forest Ecology and Management 216(1-3):257-269.

Eldridge, S.M., K.Y. Chan, Z.H. Xu, C.R. Chen, and I. Barchia. 2008. Plant-available nitrogen supply from granulated biosolids: implications for land application guidelines. Australian Journal of Soil Research 46(5):423-436.

Evanylo, G.K. 1999a. Agricultural Land Application of Biosolids in Virginia: Managing Biosolids for Agricultural Use. Crop and Soil Environmental Sciences Publication 452-303.

Evanylo, G.K. 1999b. Agricultural Land Application of Biosolids in Virginia: Production and Characteristics of Biosolids. Crop and Soil Environmental Science Publication. 452-301.

Evanylo, G.K. 2003. Effects of biosolids application timing and soil texture on nitrogen availability for corn. Communications in Soil Science and Plant Analysis 34(1-2):125-143.

Fiskell, J.G.A., D.G. Neary, and N.B. Comerford. 1990. Slash Pine and Understory Interception of Micronutrients Mineralized from Sewage-Sludge Applied to a Sandy, Acidic Forest Soil. Forest Ecology and Management 37(1-3):27-36.

Frank, J., and A.O. Stuanes. 2003. Short-term effects of liming and vitality fertilization on forest soil and nutrient leaching in a Scots pine ecosystem in Norway. Forest Ecology and Management 176:371-386.

Gilmour, J.T., C.G. Cogger, L.W. Jacobs, G.K. Evanylo, and D.M. Sullivan. 2003. Decomposition and plant-available nitrogen in biosolids: Laboratory studies, field studies, and computer simulation. Journal of Environmental Quality 32(4):1498-1507.

Gilmour, J.T., and V. Skinner. 1999. Predicting plant available nitrogen in land-applied biosolids. Journal of Environmental Quality 28(4):1122-1126.

Haith, D.A., J.E. Reynolds, P.T. Landre, and T.L. Richard. 1992. Sludge loading rates for forest land. Journal of Environmental Engineering-Asce 118(2):196-208.

Hallett, R.A., W.B. Bowden, and C.T. Smith. 1999. Nitrogen dynamics in forest soils after municipal sludge additions. Water Air and Soil Pollution 112(3-4):259-278.

Harrison, R., D.S. Xue, C. Henry, and D.W. Cole. 1994a. Long-Term effects of heavy application of biosolids on organic-matter and nutrient content of a coarse-textured forest soil. P. 165-177 in IEA/BE Workshop on Ameliorative Practices for Restoring and Maintaining Long-Term Productivity in Forests, Vaxjo, Sweden.

Harrison, R.B., S.P. Gessel, D. Zabowski, C.L. Henry, D.S. Xue, D.W. Cole, and J.E. Compton. 1994b. Mechanisms of negative impacts of three forest treatments on nutrient availability. P. 1622-1628 in Symposium on Soil and Sustained Forest Productivity, at the Annual Meeting of the ASA-CSSA-SSSA, Seattle, Wa.

Harrison, R.B., C.L. Henry, D.W. Cole, and D. Xue. 1995. Long-term changes in organic matter in soil receiving application of municipal biosolids. P. 139-153 in 8th North American Forest Soils Conference, McFee, W.W., J.M. Kelly, and J.M. Bigham (ed.), Gainsville, Fl.

Hart, J.B., P.V. Nguyen, D.H. Urie, and D.G. Brockway. 1988. Silvicultural use of waste-water sludge. Journal of Forestry 86(8):17-24.

Hartenstein, R. 1981. Sludge decomposition and stabilization. Science 212(4496):743-749.

Hattori, H., and S. Mukai. 1986. Decomposition of sewage sludges in soil as affected by their organic matter composition. Soil science and plant nutrition 32:421-432.

Henry, C., D. Sullivan, R. Rynk, K. Dorsey, and C. Cogger. 1999. Managing nitrogen from biosolids. Washington State Dept. of Ecology, [Olympia? Wash.]. 75 p.

Henry, C.L., D.W. Cole, and R.B. Harrison. 1994. Use of municipal sludge to restore and improve site productivity in forestry - The pack forest sludge research program. P. 137-149 *in* IEA/BE Workshop on Ameliorative Practices for Restoring and Maintaining Long-Term Productivity in Forests, Vaxjo, Sweden.

Johnson, D.W., and P.S. Curtis. 2000. Effects of forest management on soil C and N storage:meta analysis. Forest Ecology and Management 140(2-3):227-238.

Johnson, D.W., D.E. Todd, and V.R. Tolbert. 2003. Changes in ecosystem carbon and nitrogen in a loblolly pine plantation over the first 18 years. Soil Science Society of America Journal 67:1594-1601.

Jones, J.B., and W.J.A. Steyn. 1973. Sampling, handling, and analyzing plant tissue samples. pp. 249-270. Soil Sci. Soc. Amer., Madison, WI.

Kane, P.F. 2000. AOAC Methods. Association of Official Analytical Chemists.Washington, DC.

Kaufman, S.S., and D.A. Haith. 1986. Probabilistic Analysis of Sludge Land Application. Journal of Environmental Engineering-Asce 112(6):1041-1053.

Kelty, M.J., F.D. Menalled, and M.M. Carlton. 2004. Nitrogen dynamics and red pine growth following application of pelletized biosolids in Massachusetts, USA. Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 34(7):1477-1487.

Lal, R. 2003. Forest soils and carbon sequestration. P. 242-258 *in* 10th North American Forest Soils Conference, Saulte St Marie, Canada.

Leggett, Z.H., and D.L. Kelting. 2006. Fertilization effects on carbon pools in loblolly pine plantations on two upland sites. Soil Science Society of America Journal 70(1):279-286.

Littell, R., G. Milliken, W. Stroup, R. Wolfinger, and O. Schabenberger. 2006. SAS for Mixed Models, Second Edition. SAS Press, Cary, NC.

Luo, Y.M., and P. Christie. 2001. Short-term effects of alkaline biosolids on pH and trace metals in oligotrophic forest peat and on growth of Picea sitchensis. Forestry 74(2):145-159.

Luxmoore, R.J., M.L. Tharp, and R.A. Efroymson. 1999. Comparison of simulated forest responses to biosolids applications. Journal of Environmental Quality 28(6):1996-2007.

Magesan, G.N., C.D.A. McLay, and V.V. Lal. 1998. Nitrate leaching from a free-draining volcanic soil irrigated with municipal sewage effluent in New Zealand. Agriculture Ecosystems & Environment 70(2-3):181-187.

Maguire, R.O., J.T. Sims, S.K. Dentel, F.J. Coale, and J.T. Mah. 2001. Relationships between biosolids treatment process and soil phosphorus availability. Journal of Environmental Quality 30(3):1023-1033.

Malik, A., and J. Scullion. 1998. Soil development on restored opencase soil sites with particular reference to organic matter and aggregate stability. Soil use manage 14:234-238.

McLaren, R.G., L.M. Clucas, T.W. Speir, and A.P. van Schaik. 2007. Distribution and movement of nutrients and metals in a Pinus radiata forest soil following applications of biosolids. Environmental Pollution 147(1):32-40.

McLaren, R.G., L.M. Clucas, and M.D. Taylor. 2005. Leaching of macronutrients and metals from undisturbed soils treated with metal-spiked sewage sludge. 3. Distribution of residual metals. Australian Journal of Soil Research 43(2):159-170.

McLaren, R.G., L.M. Clucas, M.D. Taylor, and T. Hendry. 2003. Leaching of macronutrients and metals from undisturbed soils treated with metal-spiked sewage sludge. 1. Leaching of macronutrients. Australian Journal of Soil Research 41(3):571-588.

Medalie, L., W.B. Bowden, and C.T. Smith. 1994. Nutrient leaching following land application of aerobically digested municipal sewage sludge in a northern hardwood forest. J. Environ. Qual. 23(1):130-138.

Mehlich, A. 1953. Determination of P, Ca, Mg, K, Na, NH4. North Carolina Dept. of Agriculture, Agronomic Division.

Mehlich, A. 1984. Mehlich-3 soil test extractant - a modification of Mehlich-2 extractant. Communications in Soil Science and Plant Analysis 15(12):1409-1416.

Moffet, C.A., R.E. Zartman, D.B. Wester, and R.E. Sosebee. 2005. Surface biosolids application: effects on infiltration, erosion, and soil organic carbon in Chihuahuan Desert grasslands and shrublands. J Environ Qual 34(1):299-311.

Mullins, G.L., W.G. Alley, and S.B. Phillips. 2005. Sources of lime for acid soils in Virginia. 452-510. Virginia Cooperative Extention.

Nguyen, P.V., J.B. Hart, Jr., and D.M. Merkel. 1986. Municipal sludge fertilization on oak forests in Michigan: short term nutrient changes and growth responses. P 282-291 in The forest alternative for tWastewater and sludge nutreatment and utilization of municipal and industrial wastes, D.W., C., C. Henry, and N. W.L. (eds.). University of Washington Press, Seattle.WA.

Ollinger, S.V., M.L. Smith, M.E. Martin, R.A. Hallett, C.L. Goodale, and J.D. Aber. 2002. Regional variation in foliar chemistry and N cycling among forests of diverse history and composition. Ecology 83(2):339-355.

Parnaudeau, V., B. Nicolardot, and J. Pages. 2004. Relevance of organic matter fractions as predictors of wastewater sludge mineralization in soil. Journal of Environmental Quality 33(5):1885-1894.

Parnaudeau, V., B. Nicolardot, P. Robert, G. Alavoine, J. Pages, and F. Duchiron. 2006. Organic matter characteristics of food processing industry wastewaters affecting their C and N mineralization in soil incubation. Bioresource Technology 97(11):1284-1295.

Pocknee, S., and M.E. Sumner. 1997. Cation and nitrogen contents of organic matter determine its soil liming potential. Soil Science Society of America Journal 61(1):86-92.

Prescott, C.E., and L.L. Blevins. 2005. Eleven-year growth response of young conifers to biosolids or nitrogen and phosphorus fertilizer on northern Vancouver Island. Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 35(1):211-214.

Richter, D.D., D. Markewitz, J.K. Dunsomb, P.R. Heine, C.G. Wells, A.O. Stuanes, H.L. Allen, B. Urrego, K. Harrison, and G. Bonani. 1995. Carbon cycling in a loblolly pine forest: Implication for the missing carbon sink and fore the concept of soil. in Carbon forms and function in forest soil, McFee, W.W., and J.M. Kelly (eds.). Soil science society of America Inc, Gainsville, Florida.

Riekerk, H. 1978. The behavior of nutrient elements added to a forest soil with sewage sludge. Soil Science Society of America Journal 42(5):810-816.

Robinson, M.B., and P.J. Polglase. 2000. Volatilization of nitrogen from dewatered biosolids. Journal of Environmental Quality 29(4):1351-1355.

Robinson, M.B., P.J. Polglase, and C.J. Weston. 2002. Loss of mass and nitrogen from biosolids applied to a pine plantation. Australian Journal of Soil Research 40(6):1027-1039.

Robinson, M.B., and H. Roper. 2003. Volatilisation of nitrogen from land applied biosolids. Australian Journal of Soil Research 41(4):711-716.

Rostagno, C.M., and R.B. Sosebee. 2001. Surface application of biosolids in the Chihuahuan Desert: Effects on soil physical properties. Arid Land Research and Management 15(3):233-244.

Rowell, D.M., C.E. Prescott, and C.M. Preston. 2001. Decomposition and nitrogen mineralization from biosolids and other organic materials: Relationship with initial chemistry. Journal of Environmental Quality 30(4):1401-1410.

Schroder, J.L., H. Zhang, D. Zhou, N. Basta, W.R. Raun, M.E. Payton, and A. Zazulak. 2008. The effect of long-term annual application of biosolids on soil properties, phosphorus, and metals. Soil Science Society of America Journal 72(1):73-82.

Singh, R.P., and M. Agrawal. 2007. Potential benefits and risks of land application of sewage sludge. P. 347-358 *in* 30th Annual Meeting of the Society-of-General-Internal-Medicine, Toronto, Canada.

Smith, M.T.E., R.J. Smernik, G. Merrington, and M. Tibbett. 2008. Changes in sewage sludge carbon forms along a treatment stream. Chemosphere 72(6):981-985.

Smith, S.R., and E. Durham. 2000. Nitrogen release and fertiliser value of thermally-dried biosolids. CIWEM/AquaEnviro Conference:121-126.

Smith, S.R., and E. Durham. 2002. Nitrogen release and fertiliser value of thermally-dried biosolids. Journal of the Chartered Institution of Water and Environmental Management 16(2):121-126.

Smith, S.R., V. Woods, and T.D. Evans. 1998. Nitrate dynamics in biosolids-treated soils. I. Influence of biosolids type and soil type. Bioresource Technology 66:139-149.

Sommers, L.E. 1977. Chemical composition of sewage sludges and analysis of their potential use as fertilizers. Journal of Environmental Quality 6(2):225-232.

Tarrason, D., G. Ojeda, O. Ortiz, and J.M. Alcaniz. 2008. Differences on nitrogen availability in a soil amended with fresh, composted and thermally-dried sewage sludge. Bioresource Technology 99(2):252-259.

USEPA. 1983. Methods for the chemical analysis of water and wastes (MCAWW). EPA/600/4-79/020. NTIS item PB84-128677. in Environ. Monitoring and Support Lab. Office of Res. and Dev., USEPA, Cincinnati, OH.

USEPA. 2002. Test methods for evualating solid waste, physical/chemical methods SW-846 manual U.S. Environmental Protection Agency. U.S. Gov Print Office. Washington, DC.

Van Lear, D.H., and P.R. Kapeluck. 1995. Distribution of carbon in a Piedmont Soil as affected by Loblolly pine Management. in Carbon forms and functions in forest soils, McFee, W.W., and J.M. Kelly (eds.). Soil Society of America, Inc, Madison, Wisconsin, USA.

Vitousek, P.M., and P.A. Matson. 1985. Disturbance, nitrogen availability, and nitrogen losses in a intensively managed loblolly pine plantation. Ecology 66:1360-1376.

Wang, H.L., M.O. Kimberley, G.N. Magesan, R.B. McKinley, J.R. Lee, J.M. Lavery, P.D.F. Hodgkiss, T.W. Payn, P.J. Wilks, C.R. Fisher, and D.L. McConchie. 2006. Midrotation effects of biosolids application on tree growth and wood properties in a Pinus radiata plantation. Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 36(8):1921-1930.

Wang, H.L., M.O. Kimberley, and M. Schlegelmilch. 2003. Biosolids-derived nitrogen mineralization and transformation in forest soils. Journal of Environmental Quality 32(5):1851-1856.

Wang, H.L., G.N. Magesan, M.O. Kimberley, T.W. Payn, P.J. Wilks, and C.R. Fisher. 2004. Environmental and nutritional responses of a Pinus radiata plantation to biosolids application. Plant and Soil 267(1-2):255-262.

5.7. Tables and Figures

Table 5.1. Selected chemical and physical properties from the mineral soil at four different depths, from a 17-year-old loblolly pine plantation, Amelia County, VA.

Soil Depth (cm)	PH (1:1)	BD g cc^{-1}	C	N	P	K	Ca	Mg
			mg Kg^{-1}					
0-20	5.47	1.24	5700	484.0	4.0	32.0	342.0	73.0
20-40	6.18	1.39	2476	174.5	2.0	34.5	373.8	128.8
40-60	5.86	1.49	1695	165.8	2.0	31.5	334.5	129.8
60-80	5.44	1.54	1135	96.8	2.0	32.0	248.3	112.5

Table 5.2. Selected properties for biosolids surface applied in a 17-year-old loblolly pine plantation in Amelia County, VA. Biosolids source for the fall application treatment was Alexandria,VA. Biosolids sources for the spring application were Blue Plain, DC and Back River,MD , and Baltimore, MD.

Properties	Lime stabalized (Blue Plain)	Anaerobically Digested (Alexandria)	Anaerobically Digested (Back River)	Pelletized (Baltimore)
pH	12.3	8.1	8.2	5.6
		mg kg^{-1}		
Solids	352300	241500	205100	929500
Nitrogen (TKN)	31300	47500	50300	56600
Water Insol N	-	-	-	50900
Ammonia-N	1100	10200	11800	600
Phosphorus	10600	17300	20200	16100
Potassium	1300	1100	2100	2700
Sulfur	4300	10400	9300	5500
Calcium	114300	29400	22400	11200
Magnesium	2300	3300	3800	2200
Sodium	200	500	1000	400
Iron	44693	50749	55100	13682
Manganese	197	946	793	159
Copper	158	403	463	261
Zinc	314	796	867	395
Cadmium	1.2	5.8	10	-
Chromium	38	76	75	89
Nickel	16	37	36	14
Lead	38	66	66	20
Arsenic	2.9	3.6	2.1	3.9
Mercury	0.3	1.4	1.04	0.4
Selenium	2.0	3.6	4.7	2.1

Table 5.3. Final application rates and nutrient applied after of surface application of biosolids at a 17-year-old loblolly pine plantation in Amelia County, VA. Fall treatment application was anaerobically digested biosolids at a rate of 900 kg PAN ha^{-1}. Spring treatments were control, lime stabilized, pelletized, anaerobically digested biosolids, and urea + DAP fertilizer applied at a rate of 225 kg PAN ha^{-1}, and control. Anaerobically digested biosolids was also added at a rate of 900 kg PAN ha^{-1} and 1800 kg PAN ha^{-1}.

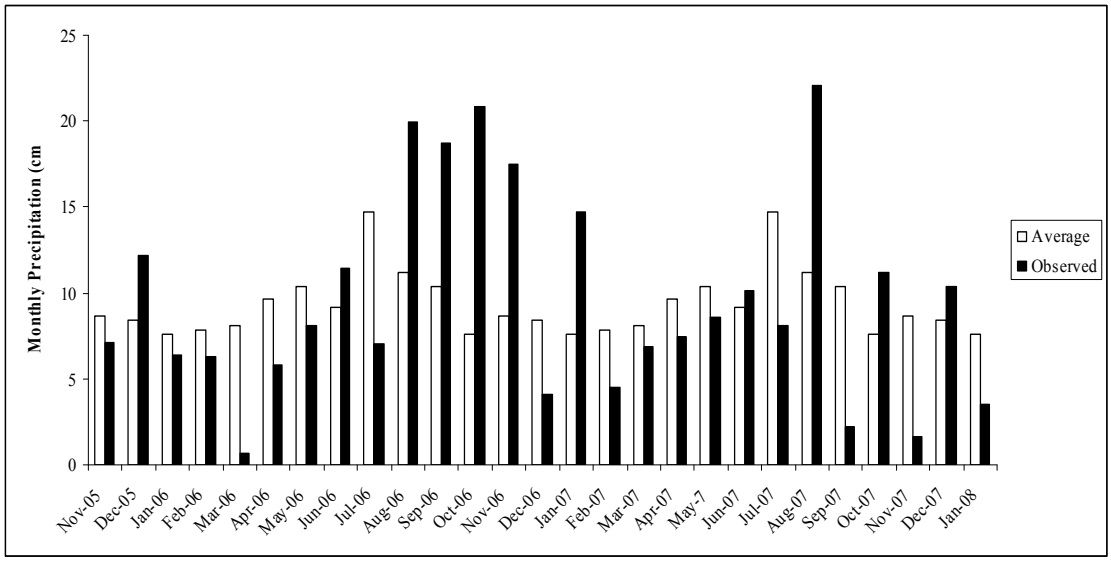
Treatments	Treatment name	Target PAN	Actual PAN	Total N	N-Org	Total P	Total K	Total Ca	Dry Weight
					Kg ha^{-1}				Mg ha^{-1}
Fall Application									
Anaer. Digested	AD900F	900	847	3202	2514	1166	74	1982	67.4
Spring Application									
Lime Stabilized	LS225	225	275	920	888	312	38	3360	29.4
Pellets	Pellet225	225	306	306	291	84	14	58	5.2
Urea + DAP	U+DAP225	225	214	214	0	23	-	-	-
Anaer. Digested	AD225	225	306	1132	866	455	47	504	22.5
Anaer. Digested	AD900S	900	860	3179	2433	1277	133	1416	63.2
Anaer. Digested	AD1800	1800	1786	6604	5055	2652	276	2941	131.3

Table 5.4. Summary of Anova for the effect of biosolids, soil depth, and time on Total N, Total C, C:N, Ca, and pH in the forest floor and the mineral soil after surface application of biosolids in a 17-year-old loblolly pine plantation in Amelia County VA.

Effect	Num Df	Total N	Total C	C:N	Ca	pH (1:1)
			(Pr>F)			
Forest Floor						
Block	3	0.5863	0.1783	0.9111	0.3617	0.8626
Treatment	7	**<.0001**	**0.0009**	**<.0001**	**<.0001**	<.0001
time	1	**0.0004**	**0.0059**	0.2775	0.3488	<.0001
Treatment*time	7	0.0692	0.4751	0.0751	0.7069	**0.0004**
Mineral Soil						
Block	3	<.0001	0.0029	0.043	0.0025	<.0001
Treatment	7	**<.0001**	0.0387	**<.0001**	0.1844	<.0001
Time	1	0.0772	0.0044	0.2312	0.4467	0.9735
Treatment*Time	7	0.2647	**0.0145**	0.1973	**0.0112**	**0.0002**
Depth	3	**<.0001**	<.0001	**0.0067**	**<.0001**	**<.0001**
Treatment*Depth	21	0.2610	0.9703	0.3304	0.875	0.4973
Depth*Time	3	0.5420	**<.0001**	0.2749	0.6148	0.6191
Treat*Depth*Time	21	0.8813	0.7513	0.9755	0.7704	0.9691

Table 5.5. Contrast for Total N, Total C, C:N, Ca, and pH from the forest floor, and mineral soil means average in a 17-year-old loblolly pine plantation following biosolids application. Treatments were anaerobically digested (AD225, AD900F, AD900, AD1800), lime stabilized (LS225), pelletized (Pellet225) biosolids, urea+DAP, and control.

Constrast	Total N	Total C	C:N	Ca	pH
			Pr >F		
Forest Floor					
Control vs AD225	0.1273	0.0598	**<.0001**	**0.0294**	**0.0017**
Control vs LS225	0.2328	**0.0363**	**0.0002**	**<.0001**	**<.0001**
Control vs Pellet225	0.1693	0.2052	**<.0001**	0.2474	**0.0026**
Control vs U+DAP225	0.8762	0.5989	0.7902	0.5285	0.3638
AD225 vs LS225	0.7231	0.808	0.0672	**0.0065**	**<.0001**
AD225 vs Pellet225	0.8710	0.5025	0.6079	0.2640	0.8782
AD225 vs U+DAP225	0.1674	0.1603	**<.0001**	**0.0072**	**0.0192**
LS225 vs U+DAP225	0.2964	0.1036	**0.0005**	**<.0001**	**<.0001**
LS225 vs Pellet225	0.8475	0.3638	**0.0231**	**0.0004**	**<.0001**
Pellet225 vs U+DAP225	0.2195	0.4479	**<.0001**	0.0813	**0.0278**
AD1800 vs AD900	**0.0165**	0.3618	0.7902	0.3927	0.5506
AD1800 vs AD225	**<.0001**	**0.0125**	**0.0003**	**0.0051**	0.1563
AD900 vs AD225	**0.0183**	0.0864	**0.0053**	**0.0352**	**0.0469**
AD900 vs AD900F	0.9693	0.959	0.9986	0.7722	0.2728
Mineral Soil					
Control vs AD225	0.3258	0.2580	0.8314	0.4939	**0.0144**
Control vs LS225	0.3540	**0.0166**	0.3502	0.2517	0.9899
Control vs Pellet225	**0.0159**	0.3934	**0.0212**	0.6152	0.6904
Control vs U+DAP225	0.0996	0.0594	0.6924	0.9863	0.6880
AD225 vs LS225	0.9551	0.1962	0.2517	0.0680	**0.0139**
AD225 vs Pellet225	0.1451	0.7796	**0.0362**	0.2360	**0.0397**
AD225 vs U+DAP225	0.5010	0.4429	0.8550	0.4832	**0.0045**
LS225 vs U+DAP225	0.4660	0.5966	0.1842	0.2588	0.6974
LS225 vs Pellet225	0.1305	0.1170	**0.0013**	0.5188	0.6811
Pellet225 vs U+DAP225	0.4293	0.2958	0.0555	0.6273	0.4241
AD1800 vs AD900	**0.0019**	0.4435	**0.0056**	0.1116	**0.0005**
AD1800 vs AD225	**<.0001**	0.8300	**<.0001**	0.2806	**<.0001**
AD900 vs AD225	0.2186	0.5807	0.1646	0.6060	0.0783
AD900 vs AD900F	0.0788	**0.0087**	**0.0037**	**0.0126**	0.0582



5.1. Average precipitation from 15-year period, (Blackstone, VA) and observed total monthly precipitation during the study duration (Amelia County, VA).

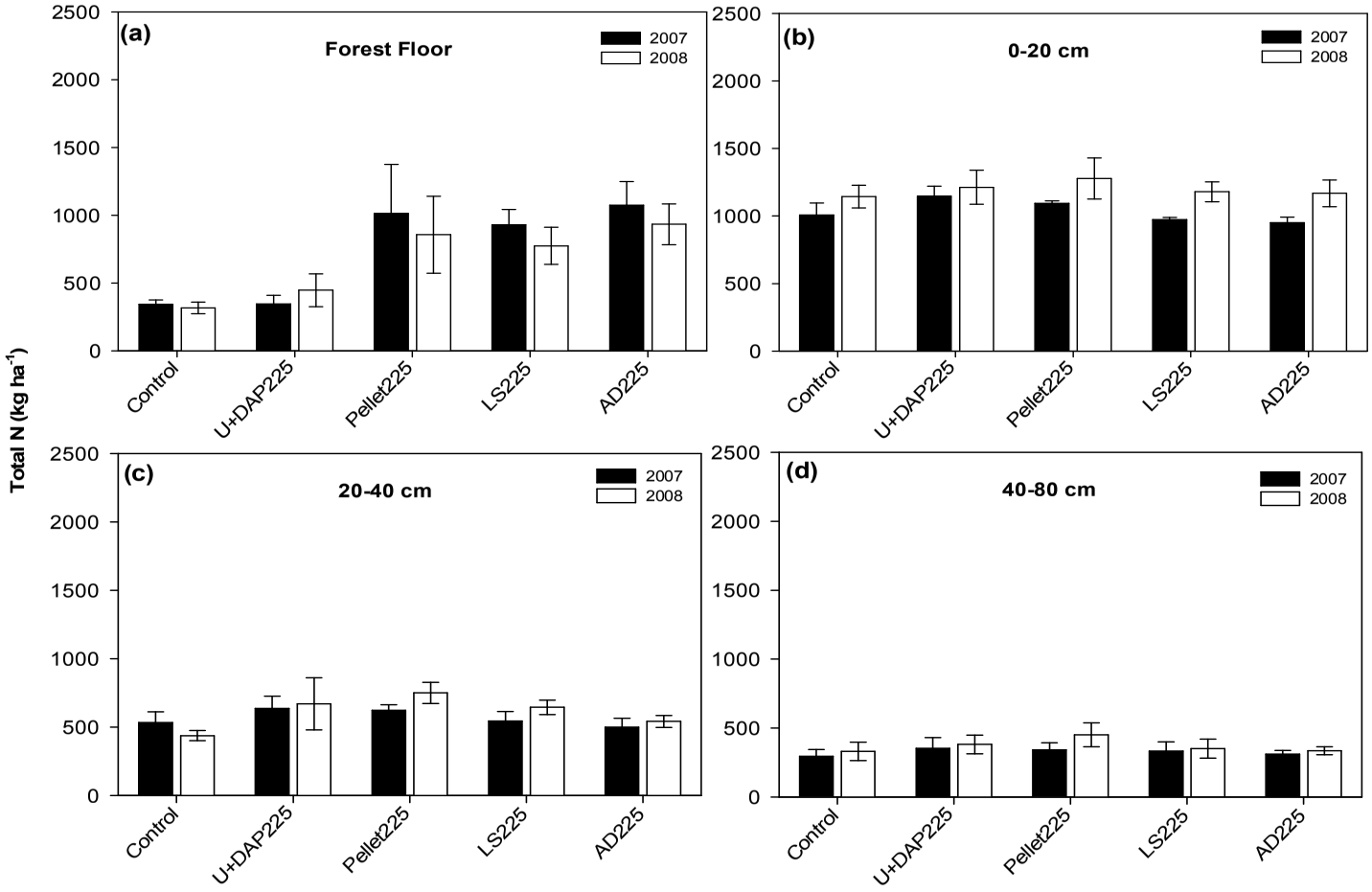


Figure 5.2. Total N from the forest floor (a), and mineral soil at different depths (b, c, d). Treatments were control, anaerobically digested (AD225) , lime stabilized (LS225), pelletized (Pellet225) biosolids, and urea+DAP applied during March 2006 at the rate of 225 kg PAN ha^{-1}. Units are in kg of N ha^{-1}. Samples were collected in February 2007, and 2008. Brackets indicate ± SE.

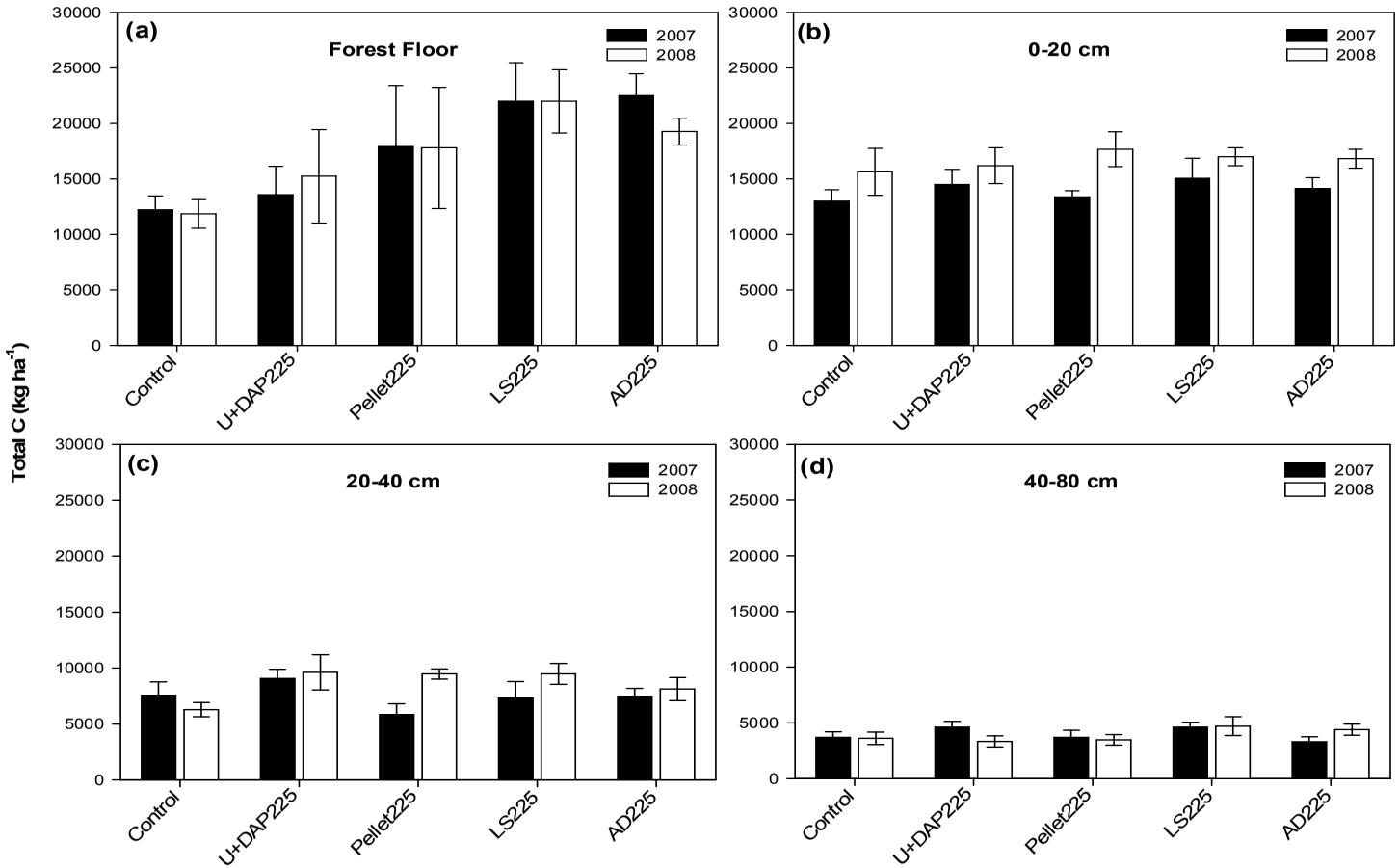


Figure 5.3. Total C from the forest floor (a), and mineral soil at different depths (b, c, d). Treatments were control, anaerobically digested (AD225), lime stabilized (LS225), pelletized (Pellet225) biosolids, and urea+DAP applied during March 2006 at the rate of 225 kg PAN ha^{-1}. Units are in kg of C ha^{-1}. Samples were collected in February 2007, and 2008. Brackets indicate ± SE.

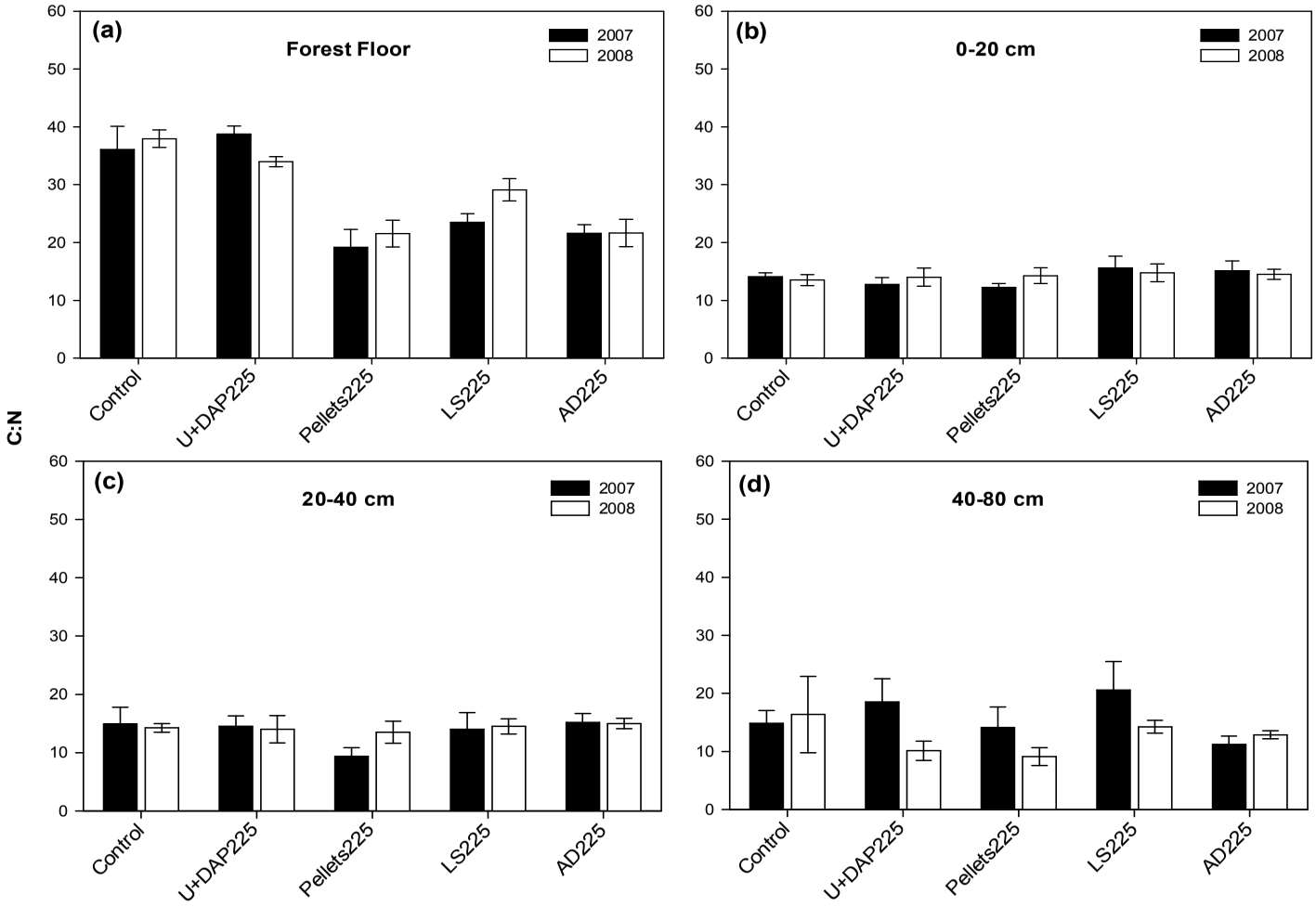


Figure 5.4. C:N from the forest floor (a), and mineral soil at different depths (b, c, d). Treatments were control, anaerobically digested (AD225), lime stabilized (LS225), pelletized (Pellet225) biosolids, and urea+DAP applied during March 2006 at the rate of 225 kg PAN ha^{-1}. Samples were collected in February 2007, and 2008. Brackets indicate ± SE.

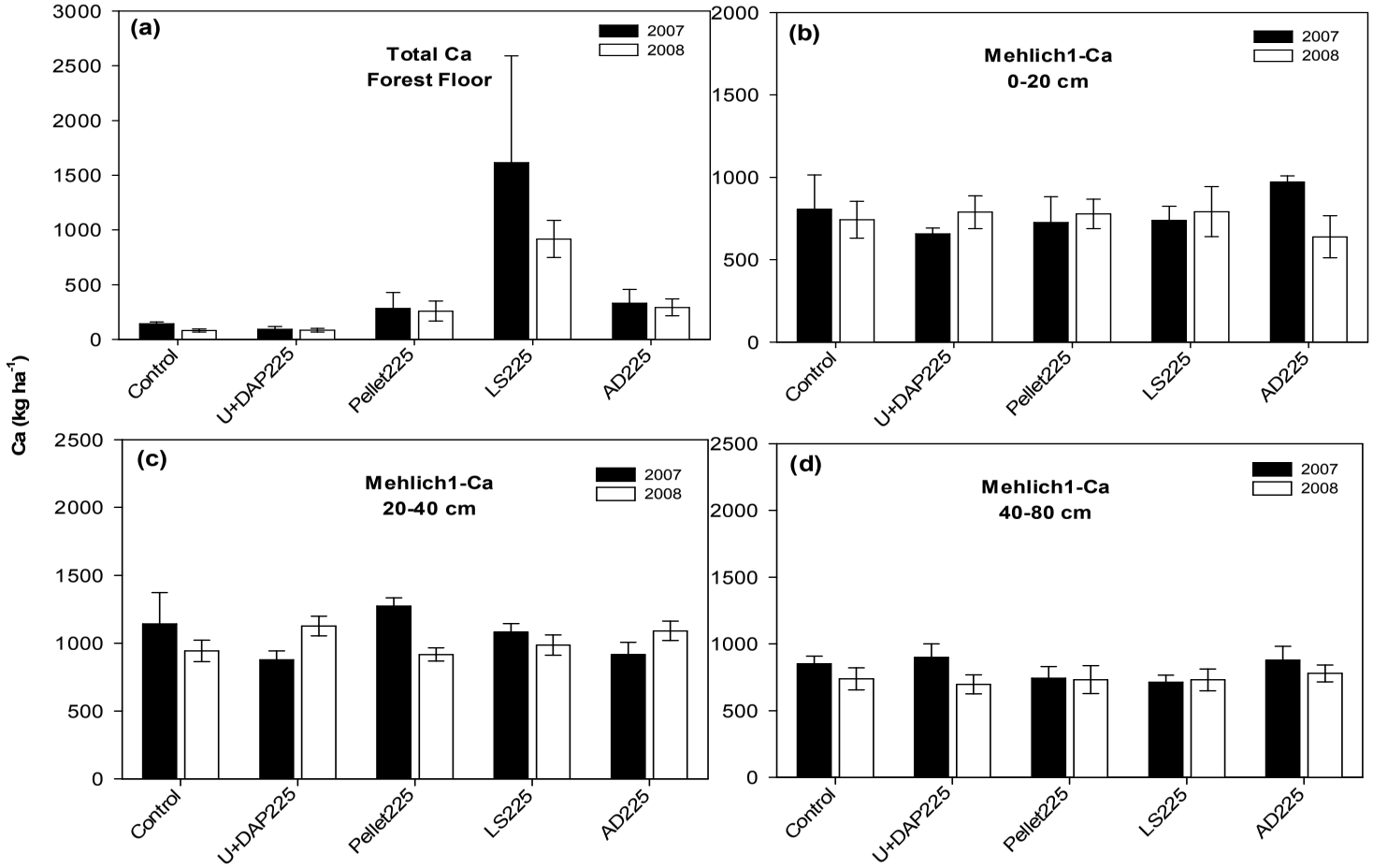


Figure 5.5. Total Ca from the forest floor (a), and extractable Ca from mineral soil at different depths (b, c, d). Treatments were control, anaerobically digested (AD225), lime stabilized (LS225), pelletized (Pellet225) biosolids, and urea+DAP applied during March 2006 at the rate of 225 kg PAN ha^{-1}. Units are in kg of Ca ha^{-1}. Samples were collected in February 2007, and 2008. Brackets indicate ± SE.

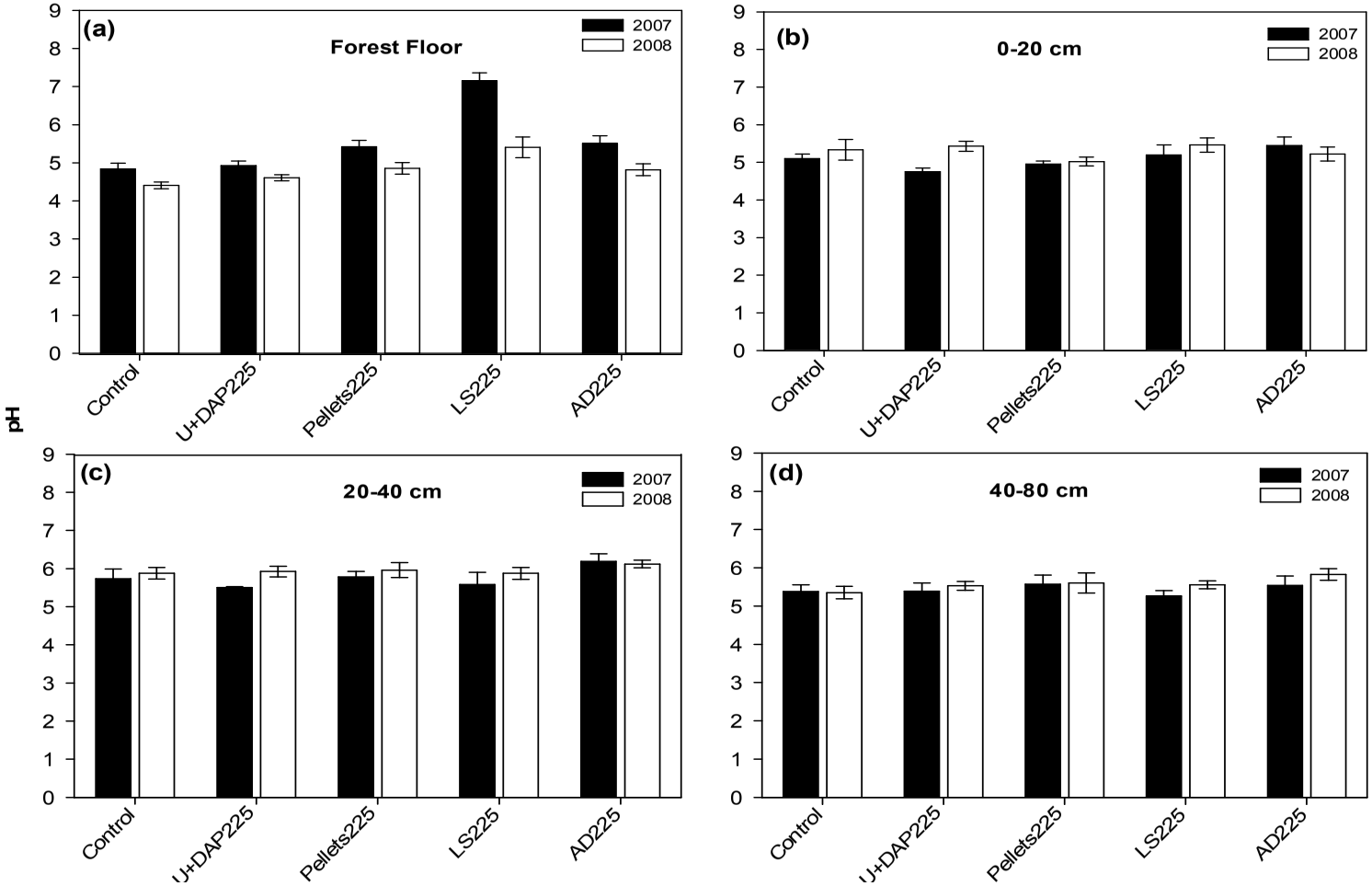


Figure 5.6. pH from the forest floor (a), and mineral soil at different depths (b, c, d). Treatments were control, anaerobically digested (AD225), lime stabilized (LS225), pelletized (Pellet225) biosolids, and urea+DAP applied during March 2006 at the rate of 225 kg PAN ha^{-1}. Samples were collected in February 2007, and 2008. Brackets indicate ± SE.

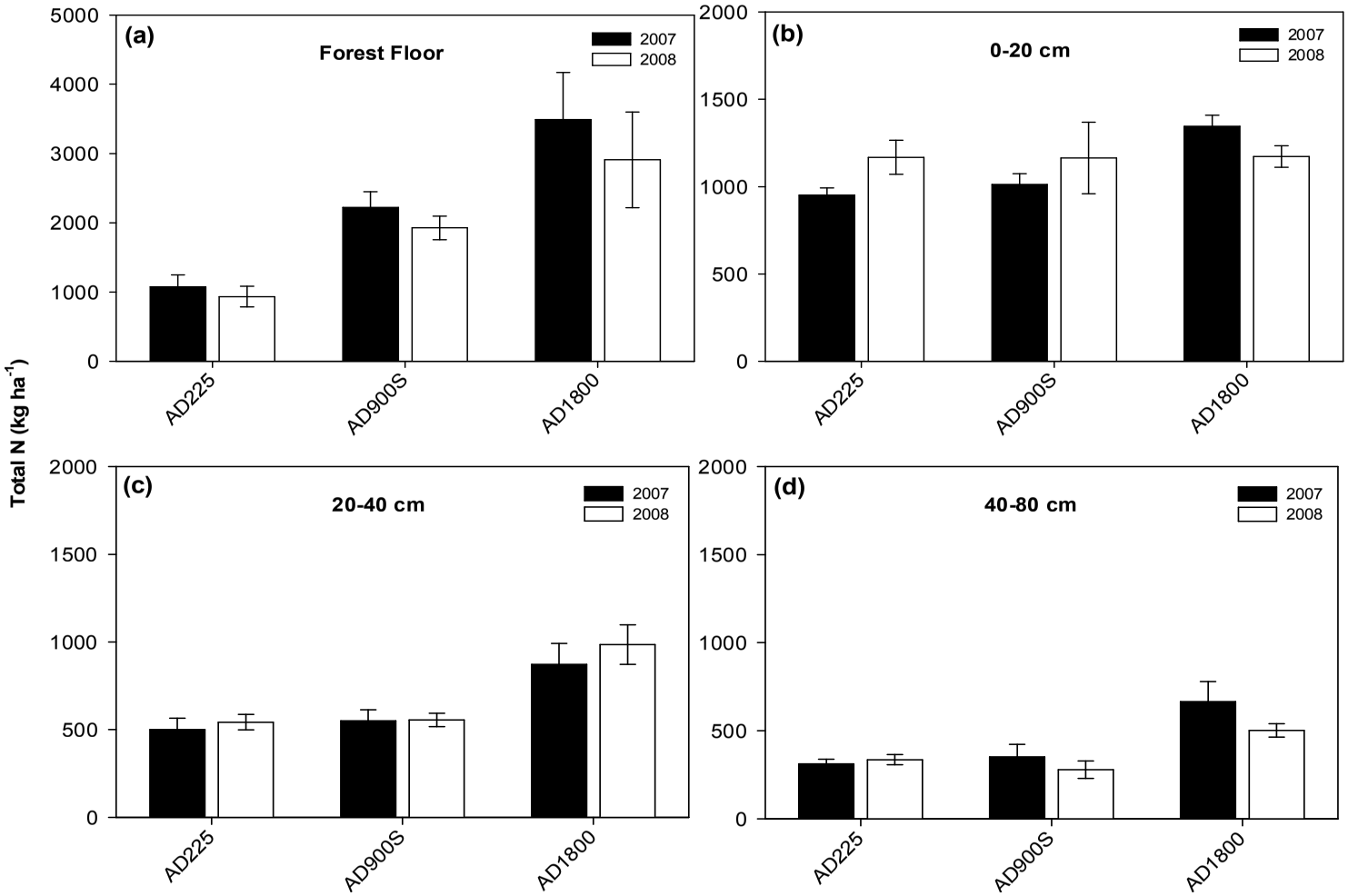


Figure 5.7. Total N from the forest floor (a), and mineral soil at different depths (b, c, d). Treatments were anaerobically digested biosolids at the rate of 225, 900, and 1800 kg PAN ha^{-1} applied during March 2006. Units are in kg of N ha^{-1}. Samples were collected in February 2007, and 2008. Forest floor and mineral soil units are in a different scale. Brackets indicate ± SE.

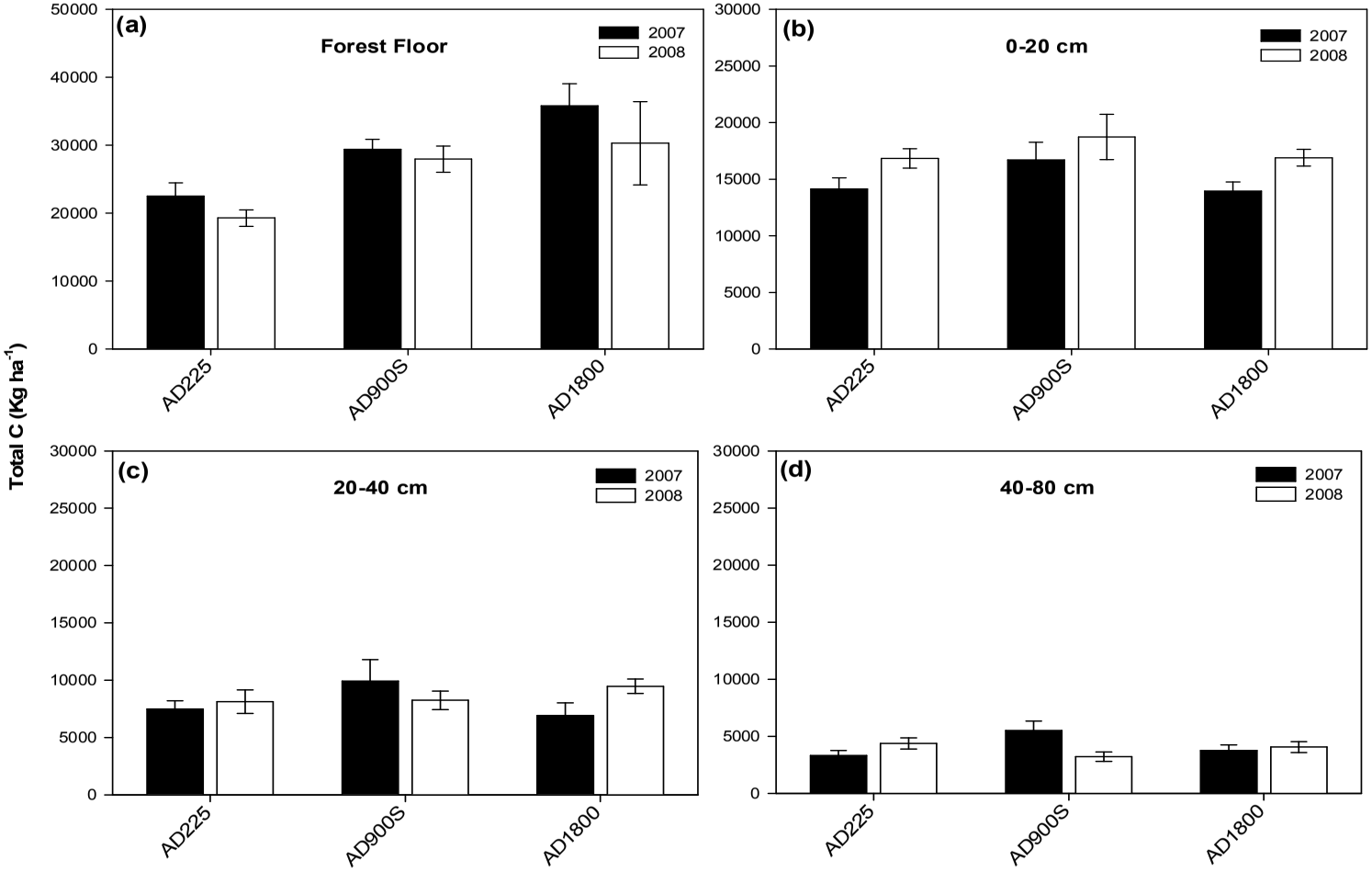


Figure 5.8. Total C from the forest floor (a), and mineral soil at different depths (b, c, d). Treatments were anaerobically digested biosolids at the rate of 225, 900, and 1800 kg PAN ha^{-1} applied during March 2006. Units are in kg of C ha^{-1}. Samples were collected in February 2007, and 2008. Forest floor and mineral soil units are in a different scale. Brackets indicate ± SE.

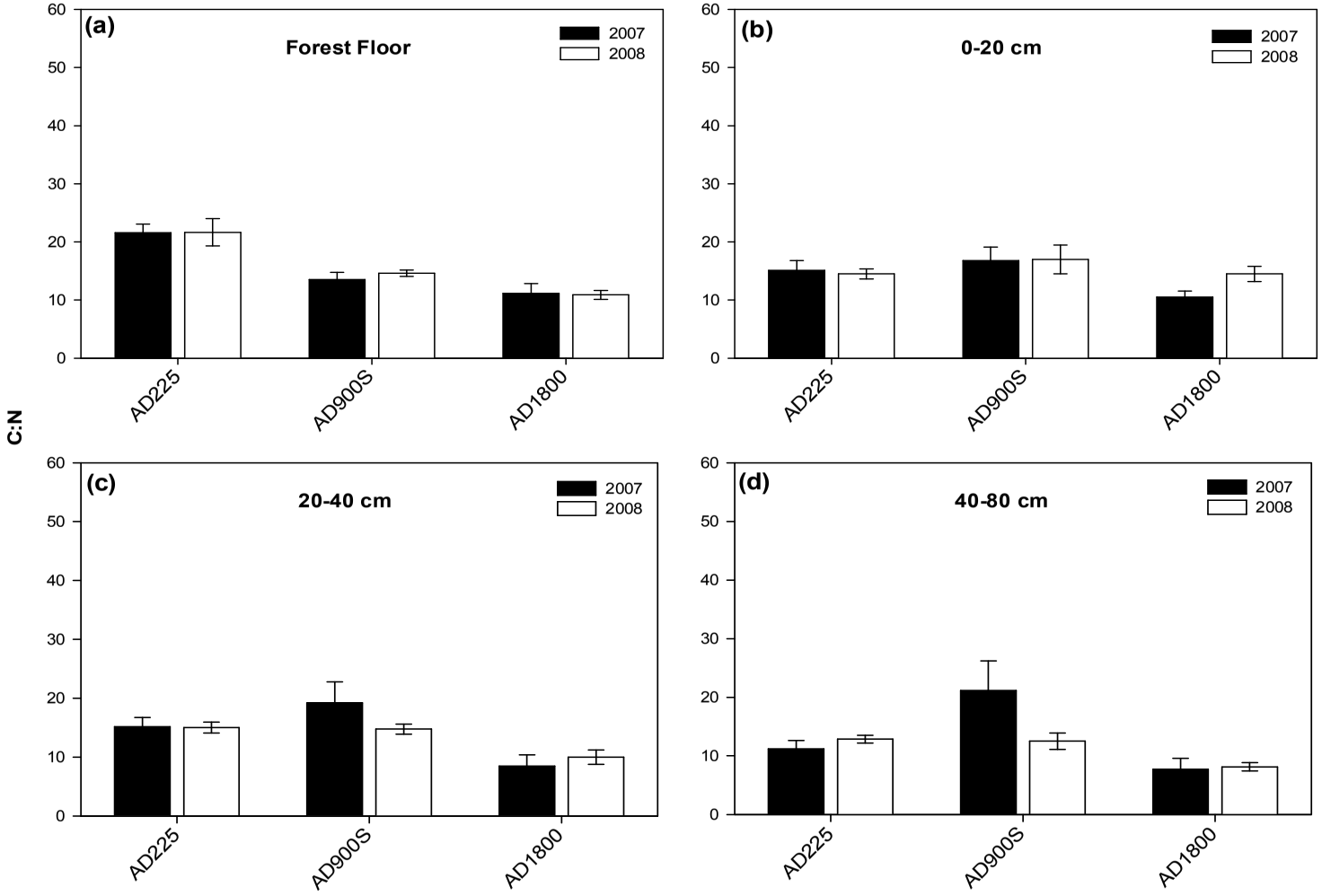


Figure 5.9. C:N from the forest floor (a), and mineral soil at different depths (b, c, d). Treatments were anaerobically digested biosolids at the rate of 225, 900, and 1800 kg PAN ha^{-1} applied during March 2006. Samples were collected in February 2007, and 2008. Forest floor and mineral soil units are in a different scale. Brackets indicate ± SE.

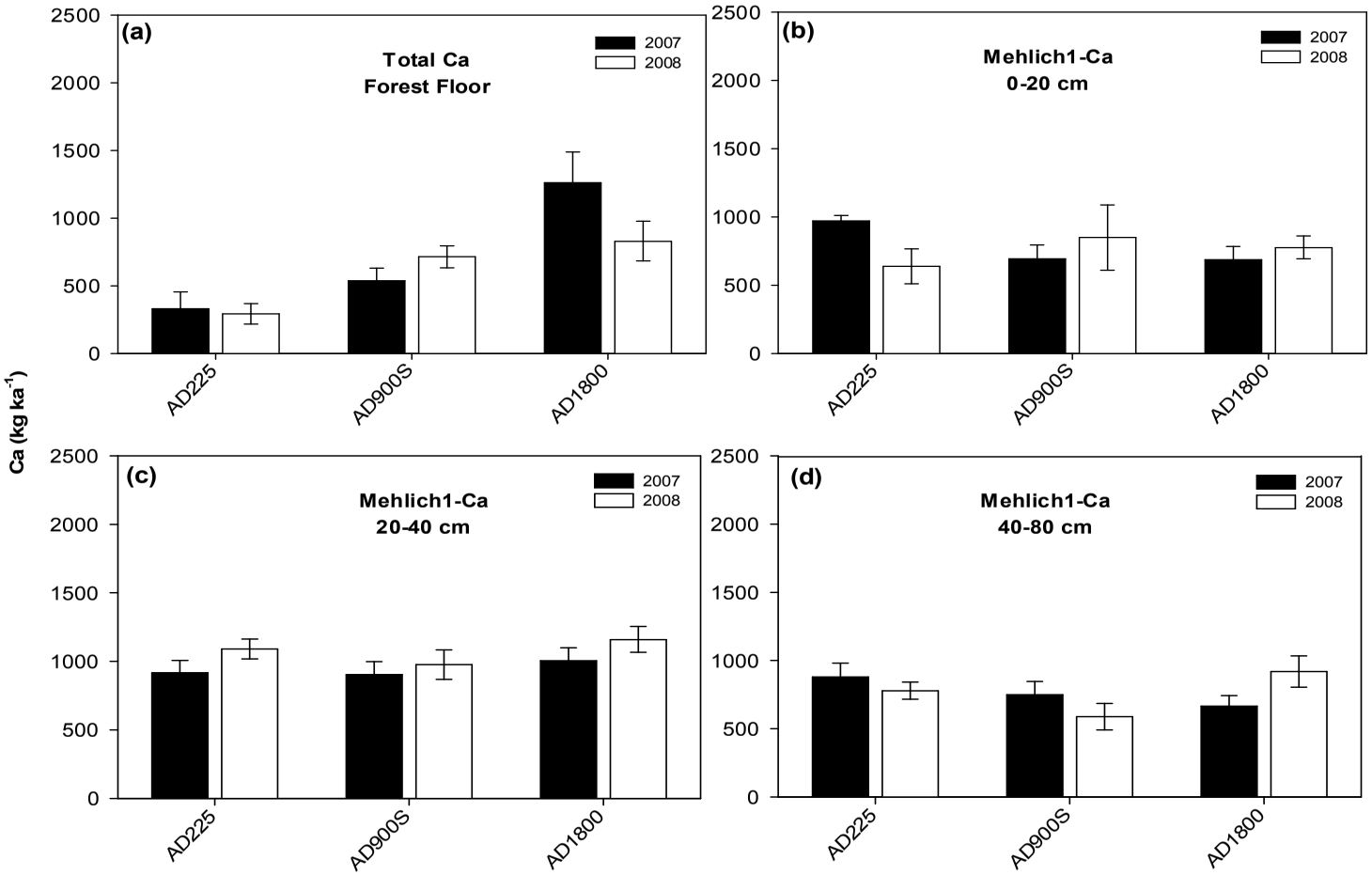


Figure 5.10. Total Ca from the forest floor (a), and extractable Ca from the mineral soil at different depths (b, c, d). Treatments were anaerobically digested biosolids at the rate of 225, 900, and 1800 kg PAN ha^{-1} applied during March 2006. Units are in kg of Ca ha^{-1}. Samples were collected in February 2007, and 2008. Forest floor and mineral soil units are in a different scale. Brackets indicate ± SE.

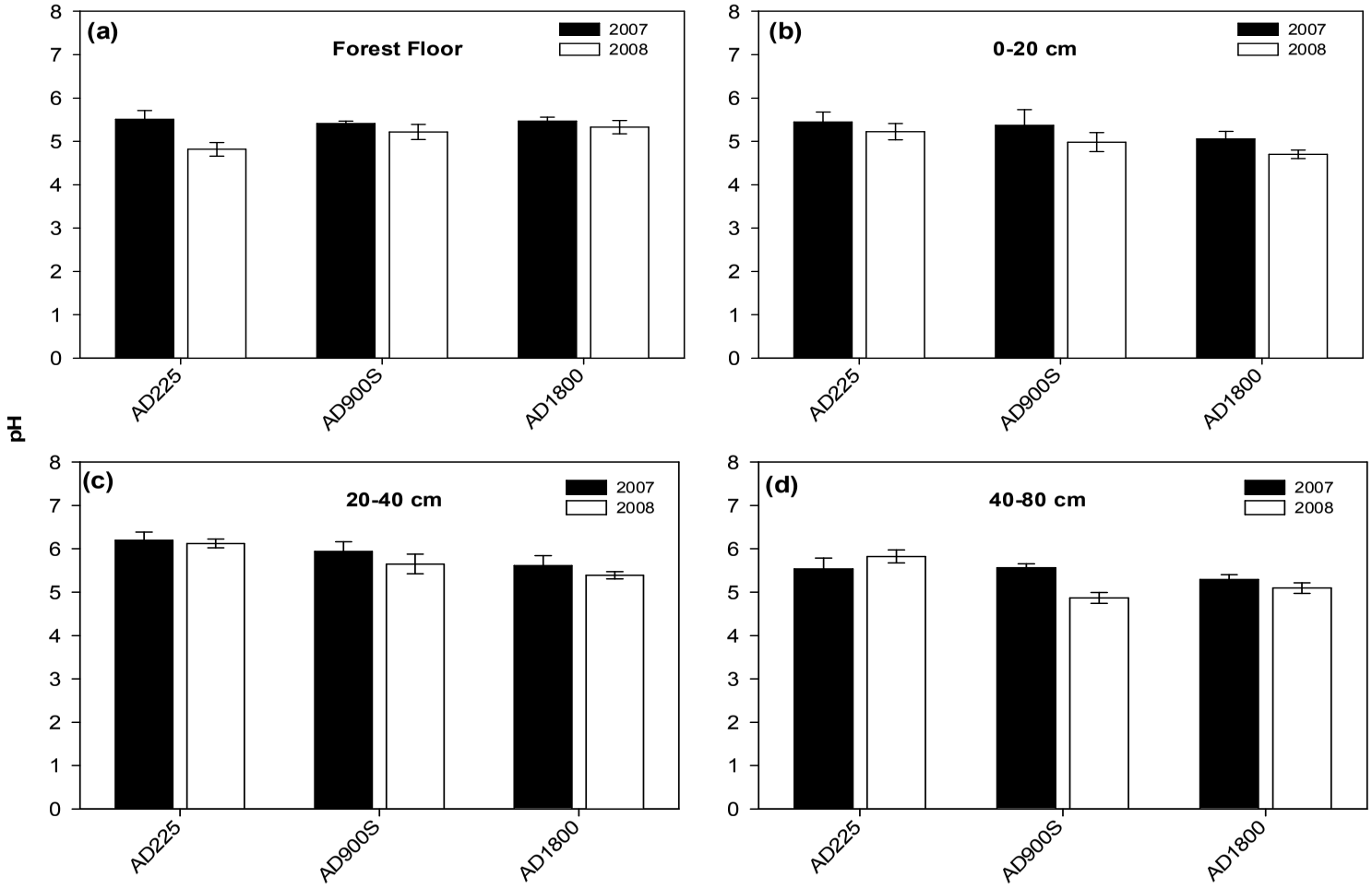


Figure 5.11. pH from the forest floor (a), and mineral soil at different depths (b, c, d). Treatments were anaerobically digested biosolids at the rates of 225, 900, and 1800 kg PAN ha^{-1} applied during March 2006. Samples were collected in February 2007, and 2008. Forest floor and mineral soil units are in a different scale. Brackets indicate ± SE.

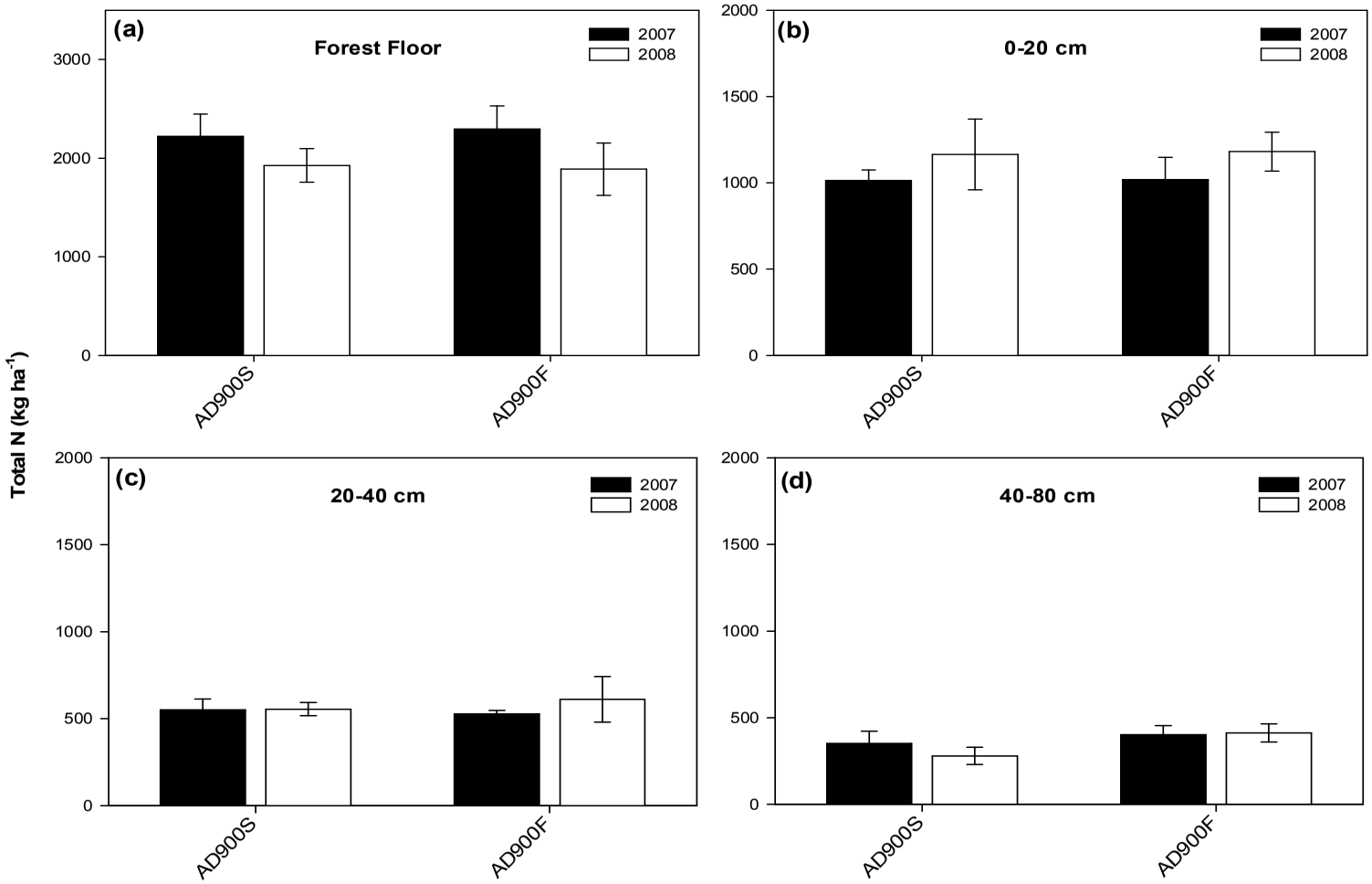


Figure 5.12. Total N from the forest floor (a) and mineral soil at different depths (b, c, d). Treatments were anaerobically digested biosolids at the rate of 900 kg PAN ha^{-1} applied November 2005 (fall), and March 2006 (spring) and reported in kg of N ha^{-1}. Samples were collected in February 2007, and 2008. Forest floor and mineral soil units are in a different scale. Brackets indicate ± SE.

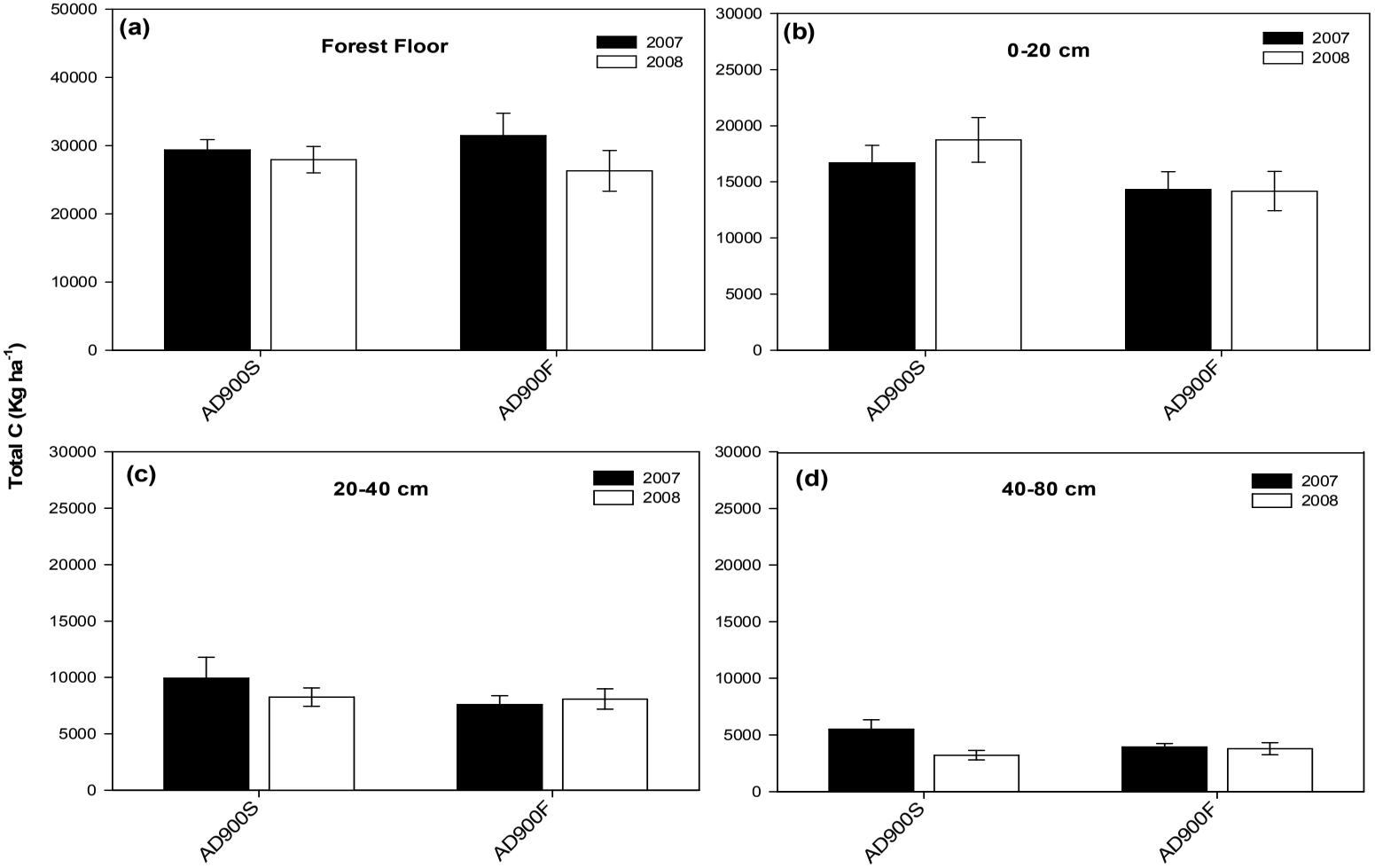


Figure 5.13. Total C from the forest floor (a) and mineral soil at different depths (b, c, d). Treatments were anaerobically digested biosolids at the rate of 900 kg PAN ha^{-1} applied November 2005 (fall), and March 2006 (spring) and reported in kg of N ha^{-1}. Samples were collected in February 2007, and 2008. Forest floor and mineral soil units are in a different scale. Brackets indicate ± SE.

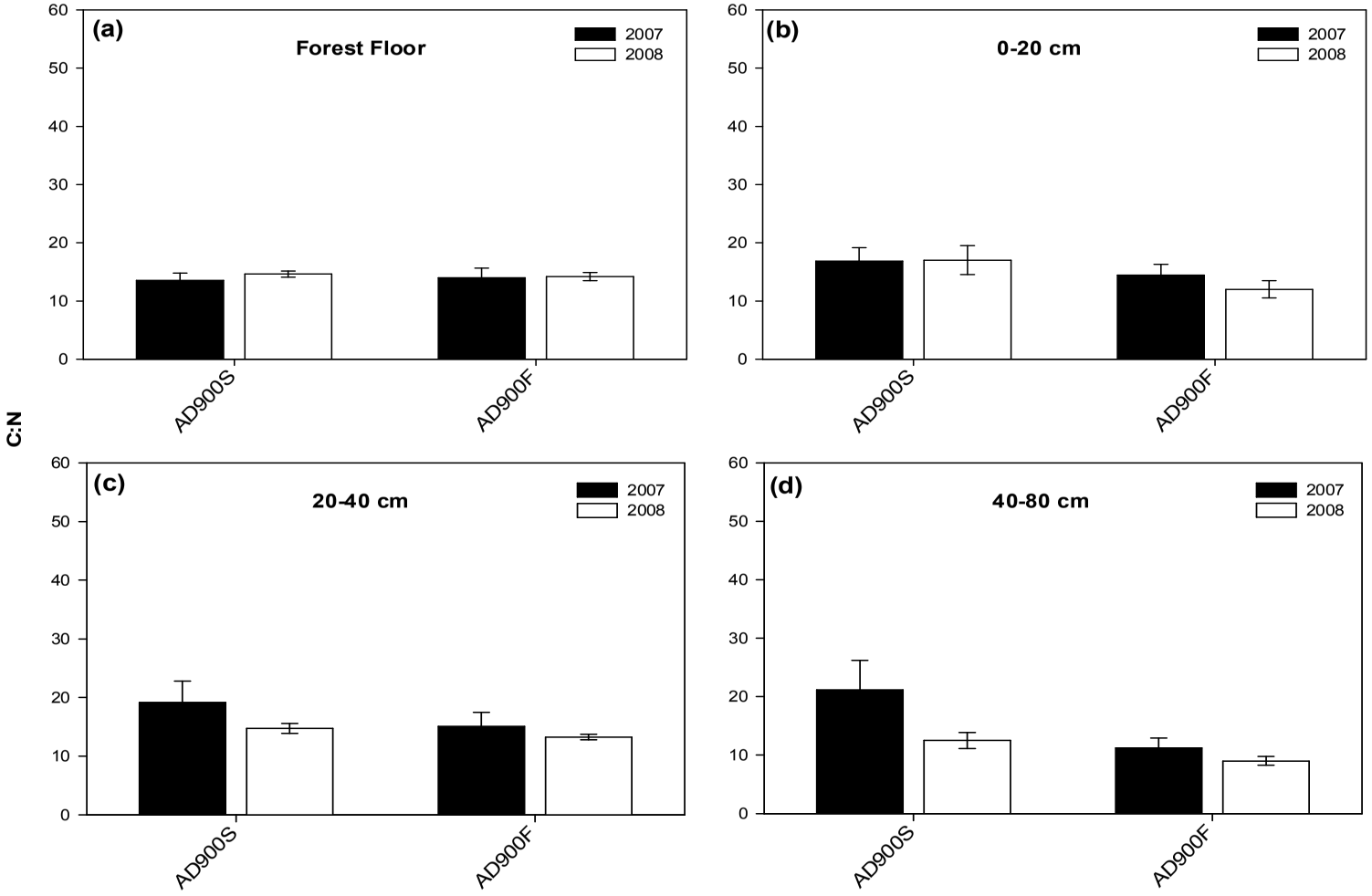


Figure 5.14. C:N from the forest floor (a) and mineral soil at different depths (b, c, d). Treatments were anaerobically digested biosolids at the rate of 900 kg PAN ha^{-1} applied November 2005 (fall), and March 2006 (spring) and reported in kg of N ha^{-1}. Samples were collected in February 2007, and 2008. Forest floor and mineral soil units are in a different scale. Brackets indicate ± SE.

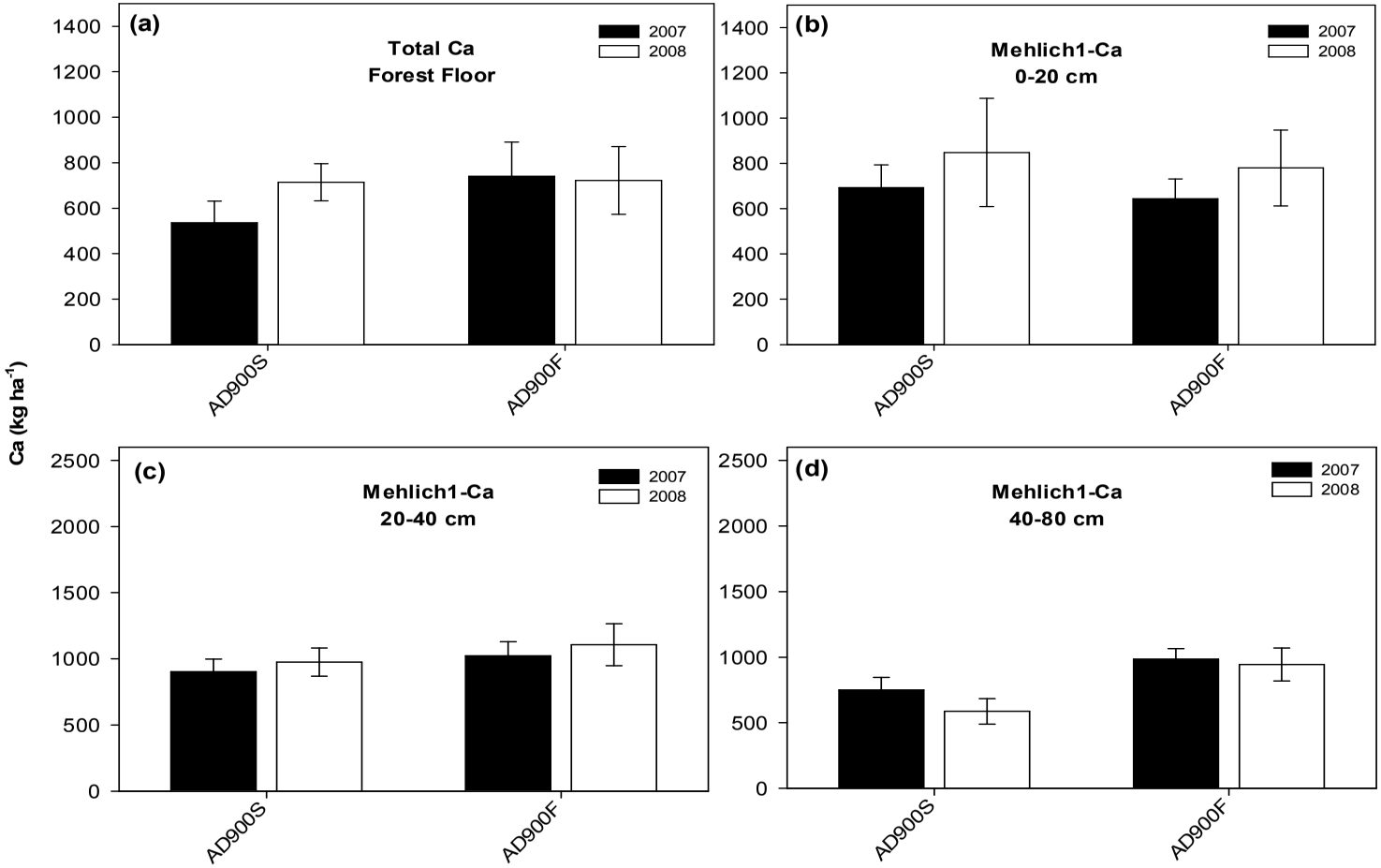


Figure 5.15. Total Ca from the forest floor (a) and extractable Ca from the mineral soil at different depths (b, c, d). Treatments were anaerobically digested biosolids at the rate of 900 kg PAN ha^{-1} applied November 2005 (fall), and March 2006 (spring) and reported in kg of N ha^{-1}. Samples were collected in February 2007, and 2008. Forest floor and mineral soil units are in a different scale. Brackets indicate ± SE.

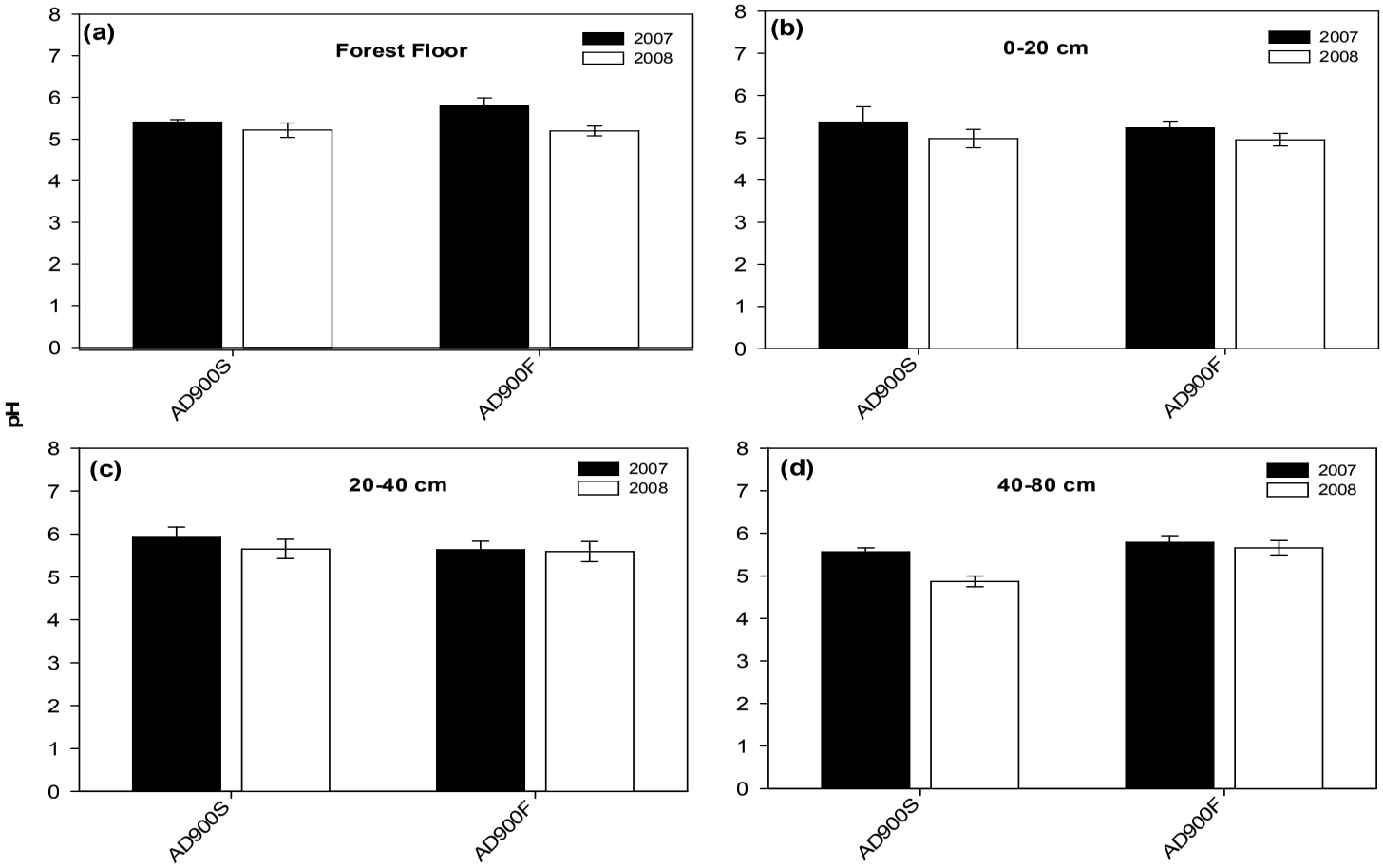


Figure 5.16. pH from the forest floor (a) and mineral soil at different depths (b, c, d). Treatments were anaerobically digested biosolids at the rate of 900 kg PAN ha^{-1} applied November 2005 (fall), and March 2006 (spring) and reported in kg of N ha^{-1}. Samples were collected in February 2007, and 2008. Forest floor and mineral soil units are in a different scale. Brackets indicate ± SE.

Chapter 6

Summary and Conclusions

6.1. Introduction

Applications of biosolids produced in wastewater treatment plants located in the Northern Virginia, Maryland, and Washington-DC corridor are largely land applied in agriculture systems as a source of nutrients. Recommendations for agriculture applications have been developed based on the amount of Nitrogen (N), Phosphorus (P), or lime required to grow a crop. However, the decreasing availability of agriculture land in the northern part of Virginia, and the large forestland available for biosolids application make forestland a good alternative for beneficial use of biosolids. In order to introduce biosolids application as an alternative source of nutrients we need to increase the understanding of the beneficial and detrimental effects of biosolids use in forests.

In the Southeast region of the USA most of loblolly pine (*Pinus taeda*) forests grow on degraded land converted from agriculture that is typically poor in available nutrients for tree growth. Fertilization prescriptions base on N and P requirements is a common forest management practices. Standard pine silviculture prescriptions incorporate the application of N and P to improve tree growth (Albaugh et al. 2007). In loblolly pine, volume growth increases of 30 % typically occur following fertilizer applications of 225 kg of N and 28 kg of P ha^{-1}(Fox et al. 2007).

Predictions of N dynamics and release from biosolids applied to forest are complicated. Soil N response following biosolids application depends on biosolids composition and forest soil

chemical, physical, and biological properties and the biosolids type. Nitrogen release from surface applied biosolids will be a function of site conditions for microbial mineralization, immobilization, vegetation coverage, plant nutrients uptake, specific properties of the organic N in the biosolids, and the time required for soil incorporation.

Several studies addressed N dynamics following application of biosolids to forest (Aschmann et al. 1992; Brockway 1983; Egiarte et al. 2005; Hallett et al. 1999; Kelty et al. 2004; Wang et al. 2004). They all recognize the potential benefits of using biosolids as a source of nutrients and promote tree growth. However, negative environmental impacts have been associated with high application rates. In the Chesapeake Bay area, there is a large concern about stream water pollution associated with excessive fertilization in agriculture and forestry. Specific evaluations comparing the interaction of different biosolids properties and how they change N availability over time in loblolly pine forest are lacking. The general objective of this research was to provide information to increase the understanding of N dynamics following biosolids application to a 17-year-old loblolly pine plantation growing in the Virginia Piedmont.

In the three previous chapters we evaluated three specific objectives looking for the effect of a single surface application of on soil N availability and mineralization (Chapter 3), N leaching (Chapter 4), and total N and C accumulation in the forest floor and mineral soil (Chapter 5). The objectives were (1) to compare the effect of the application of anaerobically digested, pelletized, lime stabilized biosolids, and urea + DAP fertilizer applied at the rate of 225 kg N ha^{-1}, (2) to compare the effect of three increasing rates (225, 900, 1800 kg N ha^{-1}) of anaerobically digested biosolids, and (3) to compare the effect of spring and fall biosolids application.

6.2. Effect of Application of Different Type of Biosolids

Our results demonstrated that surface application of different type of biosolids in a loblolly pine plantation increased soil N availability and mineralization when biosolids were applied at the permitted rate of 225 kg PAN ha^{-1} (Chapter 3 and 4). Surface soil NH_4-N and NO_3-N availability and N mineralization was significantly different among biosolids type over time (Table 3.6, 3.7, 3.9). Our results showed that N release from different type of biosolids depends on the initial inorganic N content (Fig 3.2, 4.2), and N mineralization (Fig 3.3) in biosolids. Similar N release pattern have been reported when different type of biosolids was applied to forests in other regions (Aschmann et al. 1992; Egiarte et al. 2005; Hallett et al. 1999).

The application of biosolids based on the PAN approach seems to account well for the effect of type of biosolids, since we found no differences in the average N availability and mineralization among the anaerobically digested (AD225), the lime stabilized (LS225) biosolids, and the conventional fertilizer (U+DAP225) (Table 3.5 and 4.5). However, we found that the average soil N availability and mineralization was significantly greater in the pelletized biosolids (Pellet225) treatments than in all the other treatments.

Biosolids applied in the Pellet225 and the AD225 treatments immediately increased soil N availability in comparison to biosolids in the LS225 treatment (Table 3.8 and 4.6). Field and laboratory studies recognized differences among biosolids type and showed a variable response on mineralization depending on several environmental factors and biosolids properties (Cogger et al. 2004; Donovan and Logan 1983; Epstein et al. 1978; Gale et al. 2006; Smith et al. 1998). In all the treatments, we observed a time lag between soil NH_4-N and NO_3-N concentration levels

(Fig 3.2 and 4.2). Several studies attributed this response to the growing population of nitrifiers (Garau et al. 1991; Hallett et al. 1999).

Soil N availability decreased in winter in all the treatments but remained generally higher than the control until the end of the second growing season (Fig 3.2, 3.3, and 4.2). The C:N observed in the forest floor and mineral soil following biosolids application indicated that N mineralization was still occurring in the AD225, LS225, and Pellet225 treatments at the end of the study (Table 5.5, Fig 5.4). These late N mineralization following biosolids application have been observed on field studies (Kelty et al. 2004; Robinson et al. 2002; Wang et al. 2003), and it has been attributed to the slow biosolids decomposition of he more stable organic-N forms remaining in biosolids.

Despite the increased in soil NH_4-N and NO_3-N following biosolids application at the rate of 225 kg PAN ha^{-1}, we found low NO_3-N and NH_4-N leaching in lysimeters to a depth of 80 cm (Table 4.6, 4.7). Nitrate-N concentrations in lysimeters were below water quality standard limits in all the treatments (Fig 4.2). These agree with different studies that observed low N leaching when biosolids were surface applied at a similar rate (Brockway et al. 1986; Hallett et al. 1999; Kelty et al. 2004). Surface application of biosolids reduces the potential of NO_3-N leaching.

Accumulation of N, C, and Ca in the forest floor was well correlated with the amount of biosolids applied on each treatment (Fig 5.2, 5.3 and 5.5). The average total N and C in the forest floor tended to increased following treatment application (Table 5.5).

The LS225 and AD225 treatments had larger amount of total Ca in the forest floor (Table 5.5) that directly increased pH in the forest floor during the first year (Fig 5.6). Forest floor pH

declined over time. Changes in pH after biosolids application have been attributed to the combined effect of amount of "lime" applied with biosolids (Agassi et al. 1998), and the acidification that occurred as a consequence of nitrification (Harrison et al. 1995).

The surface application of different type of biosolids had minimal impact upon total N and C in the mineral soil (Table 5.5). Differences in soil N were only temporarily in the surface mineral soil (Fig 5.2 and 5.3) (Bramryd 2002; Brockway 1983). This lack of response in the soil total N is explained by the amount of inorganic N added through biosolids in comparison to the large amount of organic N found in forest soil that make slight differences undetectable (Strahm et al. 2005).

6.3. Effect of Different Biosolids Application Rates

We also found that increasing application rates of anaerobically digested biosolids directly increased soil N availability and mineralization (Table 3.12 and 3.13). Nitrogen availability and mineralization responses lasted for a longer period of time in the AD1800 and AD900 in than in the AD225 treatment (Fig 3.4 and 3.5). With increasing loading rates of anaerobically digested biosolids soil N concentrations in solution increased over plant N requirements and soil holding capacity (Table 4.10 and 4.11). Despite the high NO_3-N retention capacity estimated with the adjusted Langmiur equation for the whole soil profile (Table 4.18), we found that NO_3-N concentration in lysimeters were above water quality standards limits during several months in the AD900 and AD1800 treatments (Fig 4.5). Our results agree with several studies that reported high levels of NO_3-N concentration in lysimeters when large rates of biosolids were applied to forest (Aschmann et al. 1990; Brockway and Urie 1983; Egiarte et al.

2005; Wells et al. 1986). Preferential flow through old root channels and soil macropores facilitated NO_3-N movement through the forest soil profile.

Significant differences in the forest floor total N, C and Ca were observed with increasing application rates of biosolids (Table 5.5, Fig 5.7, 5.8 and 5.10). Soil total N in the AD1800 treatment was significantly higher than in the AD225 and the AD900 treatments in all the sampled soil depths (Table 5.5). This indicated large N movement through the soil profile as we also found in the N leaching data in Chapter 4. The declined in soil pH in the AD1800 treatment suggested high levels of nitrification with increasing biosolids application rates (Fig 5.11). Total C accumulation was significantly higher in the forest floor in the AD1800 treatment. However, we observed no effect on soil total C with increasing application rates of biosolids (Table 5.5).

6.4. Effect of Season of Biosolids Application

We found that biosolids application during spring significantly increased soil KCl extractable N (Table 4.5), N mineralization (Table 3.5), NO_3-N leaching (Table 4.5), and total C in the mineral soil (Table 5.5) in comparison to the fall application. Fall application significantly increased NH_4-N leaching (Table 4.5) and soil extractable Ca (Table 5.5). We observed no significant effect on ion exchangeable N measured on membranes (Table 3.5), total N, C, Ca, and pH measured in the forest floor (Table 5.5), and soil total N and pH in the mineral soil (Table 5.5).

Higher temperatures occurring during spring increased N mineralization in biosolids (Fig 3.17) (Wang et al. 2003), increasing soil available N and NO_3-N movement through the soil profile (Fig 4.7). High levels of NO_3-N leaching were observed in both treatments as a consequence of the high application rates. When we paired the measurements since application

time, we observed few significant difference dates on N availability (Table 3.16) and mineralization (Table 3.17).

6.5. Incorporation of Biosolids to Forest Management

Our results demonstrated that permitted surface application of biosolids at the rate of 225 kg PAN ha^{-1} in a loblolly pine plantation increased surface soil N availability without increasing the potential for NO_3-N groundwater pollution. Despite similar application rates based on PAN, we observed that soil N availability and mineralization in the Pellet225 was significantly greater than the AD225, LS225, and the U+DAP225 treatments. These results were likely a function of the variable N release through mineralization in biosolids.

Forest applications of pelletized biosolids have also the advantage of reducing the volume of transported material to the site and odors emission, with minimal impact on communities close to the site since they are applied as a regular slow release fertilizer. However, operational use of pelletized biosolids could be more restrictive, since only anaerobically digested and lime stabilized biosolids are provided and distributed to forest landowners for free by the wastewater treatments facilities that operate in Virginia. Loblolly pine forests in Virginia are located relatively far from populated areas, and if anaerobically digested or lime stabilized biosolids are correctly applied odors and transportation problems should be minimized in comparison to agriculture land applications. Additionally, knowledge of use of different biosolids type as a source of N in pine plantations in Virginia is as important as knowing the impact of biosolids on water quality, wildlife, and understory vegetation.

Surface application of biosolids showed little potential to increase soil C sequestration. When biosolids were surface applied to loblolly pine forest, we observed C accumulated in the

forest floor but no effect on soil C in any of the biosolids type or rate treatments. On the other hand, with high application rates of biosolids N availability increased above plant requirements and soil sorption capacity, increasing N leaching and the potential for groundwater pollution. A system for predicting N release from different type of biosolids under forest operational conditions is needed to develop recommendations rates base on PAN and forest site characteristics. Our results suggested that application rates of anaerobically digested biosolids could be higher than 225 kg PAN ha^{-1} but bellow the 900 kg PAN ha^{-1}. However, biosolids application systems to pine forests in the Piedmont need to account for the soil variability and biosolids type, since biosolids application programs in Virginia also regulate P applications.

There are still important questions that need to be address to maximize the amount of biosolids applied to forest and tree growth, minimizing detrimental consequences. From the forest management point of view, further investigations should look for the long-term effect of biosolids application on soil and water quality, wildlife, other nutrients accumulation, and how biosolids applications affect pine productivity.

6.6. References

Agassi, M., W.F.A. Kirsten, A.H. Loock, and P. Fine. 1998. Percolation and leachate composition in a disturbed soil layer mulched with sewage biosolids. Soil & Tillage Research 45(3-4):359-372.

Albaugh, T.J., H.L. Allen, and T.R. Fox. 2007. Historical patterns of forest fertilization in the southeastern United States from 1969 to 2004. Southern Journal of Applied Forestry 31(3):129-137.

Aschmann, S.G., M.S. McIntosh, J.S. Angle, and R.L. Hill. 1992. Nitrogen movement under a hardwood forest amendd with liquid waste-water sludge. Agriculture Ecosystems & Environment 38(4):249-263.

Aschmann, S.G., M.S. McIntosh, J.S. Angle, R.L. Hill, and R.R. Weil. 1990. Nitrogen status of forest floor, soils, and vegetation following municipal waste-water sludge application. Journal of Environmental Quality 19(4):687-694.

Bramryd, T. 2002. Impact of sewage sludge application on the long-term nutrient balance in acid soils of Scots pine (Pinus sylvestris, L.) forests. Water Air and Soil Pollution 140(1-4):381-399.

Brockway, D.G. 1983. Forest Floor, Soil, and vegetation responses to sludge fertilization in Red and White-Pine Plantations. Soil Science Society of America Journal 47(4):776-784.

Brockway, D.G., and D.H. Urie. 1983. Determining Sludge Fertilization Rates for Forests from Nitrate-N in Leachate and Groundwater. Journal of Environmental Quality 12(4):487-492.

Brockway, D.G., D.H. Urie, P.V. Nguyen, and J.B. Hart. 1986. Wastewater and sludge nutrient utilization in forest ecosystems. P. 221-245 in The forest alternative for Wastewater and sludge treatment and utilization of municipal and industrial wastes, D.W., C., C. Henry, and N. W.L. (eds.). University of Washington Press, Seattle.WA.

Cogger, C.G., A.I. Bary, D.M. Sullivan, and E.A. Myhre. 2004. Biosolids processing effects on first- and second-year available nitrogen. Soil Science Society of America Journal 68(1):162-167.

Donovan, W.C., and T.J. Logan. 1983. Factors affecting ammonia volatilization from sewage-sludge applied to soil in a laboratory study. Journal of Environmental Quality 12(4):584-590.

Egiarte, G., M.C. Arbestain, A. Alonsoa, E. Ruiz-Romera, and M. Pinto. 2005. Effect of repeated applications of sewage sludge on the fate of N in soils under Monterey pine stands. Forest Ecology and Management 216(1-3):257-269.

Epstein, E., D.B. Keane, J.J. Meisinger, and J.O. Legg. 1978. Mineralization of nitrogen from sewage sludge and sludge compost. Journal of Environmental Quality 7(2):217-221.

Fox, T.R., H.L. Allen, T.J. Albaugh, R. Rubilar, and C.A. Carlson. 2007. Tree nutrition and forest fertilization of pine plantations in the southern United States. Southern Journal of Applied Forestry 31(1):5-11.

Gale, E.S., D.M. Sullivan, C.G. Cogger, A.I. Bary, D.D. Hemphill, and E.A. Myhre. 2006. Estimating plant-available nitrogen release from manures, composts, and specialty products. Journal of Environmental Quality 35(6):2321-2332.

Garau, M.A., J.L. Dalmau, and M.T. Felipo. 1991. Nitrogen Mineralization in Soil Amended with Sewage-Sludge and Fly-Ash. Biology and Fertility of Soils 12(3):199-201.

Hallett, R.A., W.B. Bowden, and C.T. Smith. 1999. Nitrogen dynamics in forest soils after municipal sludge additions. Water Air and Soil Pollution 112(3-4):259-278.

Harrison, R.B., C.L. Henry, D.W. Cole, and D. Xue. 1995. Long-term changes in organic matter in soil receiving application of municipal biosolids. P. 139-153 in 8th North American Forest Soils Conference, McFee, W.W., J.M. Kelly, and J.M. Bigham (eds.), Gainsville, Fl.

Kelty, M.J., F.D. Menalled, and M.M. Carlton. 2004. Nitrogen dynamics and red pine growth following application of pelletized biosolids in Massachusetts, USA. Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 34(7):1477-1487.

Medalie, L., W.B. Bowden, and C.T. Smith. 1994. Nutrient Leaching Following Land Application of Aerobically Digested Municipal Sewage-Sludge in a Northern Hardwood Forest. Journal of Environmental Quality 23(1):130-138.

Robinson, M.B., P.J. Polglase, and C.J. Weston. 2002. Loss of mass and nitrogen from biosolids applied to a pine plantation. Australian Journal of Soil Research 40(6):1027-1039.

Robinson, M.B., and H. Roper. 2003. Volatilisation of nitrogen from land applied biosolids. Australian Journal of Soil Research 41(4):711-716.

Smith, S.R., V. Woods, and T.D. Evans. 1998. Nitrate dynamics in biosolids-treated soils. I. Influence of biosolids type and soil type. Bioresource Technology 66:139-149.

Sommers, L.E. 1977. Chemical composition of sewage sludges and analysis of their potential use as fertilizers. Journal of Environmental Quality 6(2):225-232.

Strahm, B.D., R.B. Harrison, T.A. Terry, B.L. Flaming, C.W. Licata, and K.S. Petersen. 2005. Soil solution nitrogen concentrations and leaching rates as influenced by organic matter retention on a highly productive Douglas-fir site. Forest Ecology and Management 218(1-3):74-88.

Wang, H., G.N. Magesan, P.W. Clinton, and J.M. Lavery. 2004. Using natural N-15 abundances to trace the fate of waste-derived nitrogen in forest ecosystems: New Zealand case studies. P. 31-38 in 4th International Conference on Applications of Stable Isotope Techniques to Ecological Studies, Wellington, New Zealand.

Wang, H.L., M.O. Kimberley, and M. Schlegelmilch. 2003. Biosolids-derived nitrogen mineralization and transformation in forest soils. Journal of Environmental Quality 32(5):1851-1856.

Wells, C.G., C.E. Murphy, C. Davis, D.M. Stone, and G.J. Hollod. 1986. Effect of sewage sludge from two sources on element flux in soil solution of loblolly pine plantations. P. 154-165 in The Forest Alternative for Treatment and Utilization of Municipal and Industrial Wastes, Cole, D.W., C. Henry, and W.L. Nutter (eds.). University of Washington Press, Seattle and London.

List of References

Agassi, M., W.F.A. Kirsten, A.H. Loock, and P. Fine. 1998. Percolation and leachate composition in a disturbed soil layer mulched with sewage biosolids. Soil & Tillage Research 45(3-4):359-372.

Albaugh, T.J., H.L. Allen, and T.R. Fox. 2007. Historical patterns of forest fertilization in the southeastern United States from 1969 to 2004. Southern Journal of Applied Forestry 31(3):129-137.

APHA. 1998. Standard methods for examination of water and wastewater. Am. Water Works Assoc., and Water Environment Federation, Washington, DC.

Aschmann, S.G., M.S. McIntosh, J.S. Angle, and R.L. Hill. 1992. Nitrogen movement under a hardwood forest amendd with liquid waste-water sludge. Agriculture Ecosystems & Environment 38(4):249-263.

Aschmann, S.G., M.S. McIntosh, J.S. Angle, R.L. Hill, and R.R. Weil. 1990. Nitrogen status of forest floor, soils, and vegetation following municipal waste-water sludge application. Journal of Environmental Quality 19(4):687-694.

Aust, W.M. 1994. Best management practices for forested wetlands in the southern Appalachian region. Water, Air, and Soil Pollution 77:457-468.

Barbarika, A., L.J. Sikora, and D. Colacicco. 1985. Factors affecting mineralization of nitrogen in sewage-sludge applied to soils. Soil Science Society of America Journal 49(6):1403-1406.

Basta, N.T. 1995. Land application of biosolids : a review of research concerning benefits, environmental impacts, and regulations of applying treated sewage sludge. Oklahoma Agricultural Experiment Station : Center for Agriculture and the Environment, Division of Agricultural Sciences and Natural Resources, Oklahoma State University, [Stillwater]. xii, 59 p. p.

Beauchamp, E.G., G.E. Kidd, and G. Thurtell. 1978. Ammonia volatilization from sewage sludge applied in field. Journal of Environmental Quality 7(1):141-146.

Beauchamp, E.G., Y.K. Soon, and J.R. Moyer. 1979. Nitrate Production from Chemically Treated Sewage Sludges in Soil. Journal of Environmental Quality 8(4):557-560.

Beech, N., K. Crawford, N. Goldstein, G. Kester, M. Lono-Batura, and E. Dzieyk. 2007. A national biosolids regulation, quality, end use and disposal survey: Final report. North East Biosolids and Residuals Association (NEBRA). 30.

Bengtsson, G., P. Bengtson, and K.F. Mansson. 2003. Gross nitrogen mineralization-, immobilization-, and nitrification rates as a function of soil C/N ratio and microbial activity. Soil Biology & Biochemistry 35(1):143-154.

Berry, C.R. 1987. Use of municipal sewage sludge for improvement of forest sites in the southeast. United Stated Department of Agriculture, Forest Service. 33 pp.

Binkley, D., H. Burnham, and H.L. Allen. 1999. Water quality impacts of forest fertilization with nitrogen and phosphorus. Forest Ecology and Management 121(3):191-213.

Boesch, D.F., R.B. Brinsfield, and R.E. Magnien. 2001. Chesapeake Bay eutrophication: Scientific understanding, ecosystem restoration, and challenges for agriculture. P. 303-320 in Annual Meetings of the American-Society-of-Agronomy, Anaheim, California.

Bosatta, E., and F. Berendse. 1984. Energy or nutrients regulation of decomposition-Implications for the mineralization immobilization response to pertubations. Soil Biology & Biochemistry 16(1):63-67.

Bowden, W., and M.J. Hann. 1997. The availability of nitrogen following topsoil application of liquid digested sludge. Nutrient Cycling in Agroecosystems 47(2):167-172.

Boyle, M., and E.A. Paul. 1989a. Carbon and nitrogen mineralization kinetics in soil previously amended with sewage-sludge. Soil Science Society of America Journal 53(1):99-103.

Boyle, M., and E.A. Paul. 1989b. Nitrogen transformations in soils previously amended with sewage-sludge. Soil Science Society of America Journal 53(3):740-744.

Boyle, M. 1990. Biodegradation of land-applied sludge. Journal of Environmental Quality 19(4):640-644.

Bramryd, T. 1981. Comparative studies of nitrogen mineralization in forest soils fertilized with fluid and dewatered sewage sludge. P. 475-483.

Bramryd, T. 2002. Impact of sewage sludge application on the long-term nutrient balance in acid soils of Scots pine (Pinus sylvestris, L.) forests. Water Air and Soil Pollution 140(1-4):381-399.

Brockway, D.G. 1983. Forest Floor, Soil, and vegetation responses to sludge fertilization in Red and White-Pine Plantations. Soil Science Society of America Journal 47(4):776-784.

Brockway, D.G., and D.H. Urie. 1983. Determining Sludge Fertilization Rates for Forests from Nitrate-N in Leachate and Groundwater. Journal of Environmental Quality 12(4):487-492.

Brockway, D.G., D.H. Urie, P.V. Nguyen, and J.B. Hart. 1986. Wastewater and sludge nutrient utilization in forest ecosystems. P. 221-245. in The forest alternative for wastewater and sludge

nutreatment and utilization of municipal and industrial wastes, D.W., C., C. Henry, and N. W.L. (eds.). University of Washington Press, Seattle.WA.

Brown, D. 2004. Building carbon credits with biosolids recycling. BioCycle: Journal of Composting & Organic Recycling. September:25-29.

Brown, S., and C. Henry. 2000. Using biosolids at mine reclamation sites. BioCycle: Journal of Composting & Organic Recycling 41(2):18-19.

Brumme, R., and P.K. Khanna. 2008. Ecological and site historical aspects of N dynamics and current N status in temperate forests. Global Change Biology 14(1):125-141.

Burton, A.J., J.B. Hart, and D.H. Urie. 1990. Nitrification in slude-amended michigan forest soils. Journal of Environmental Quality 19(3):609-616.

Cabrera, M.L., D.E. Kissel, and M.F. Vigil. 2005. Nitrogen mineralization from organic residues: Research opportunities. Conference on Sustainable Land Application:75-79.

Carter, M.C., T.J. Dean, M. Zhuo, M.G. Messina, and Z. Wang. 2002. Short-term changes in soil C, N, and biota following harvesting and regeneration of loblolly pine (Pinus taeda L.). Forest Ecology and Management 164:67-88.

Chae, Y.M., and M.A. Tabatabai. 1986. Mineralization of nitrogen in soils amended with organic wastes. Journal of Environmental Quality 15(2):193-198.

Chapman-King, R., T.M. Hinckley, and C.C. Grier. 1986. Growth response of forest trees to wastewater and sludge application. P. 209-220.

Cogger, C.G., A.I. Bary, D.M. Sullivan, and E.A. Myhre. 2004. Biosolids processing effects on first- and second-year available nitrogen. Soil Science Society of America Journal 68(1):162-167.

Cooperband, L.R., and T.J. Logan. 1994. Measuring in-Situ Changes in Labile Soil-Phosphorus with Anion-Exchange Membranes. Soil Science Society of America Journal 58(1):105-114.

Corey, J.C., M.W. Lower, and C.E. Davis. 1986. The sludge application program at the Savannah River plant. in The Forest Alternative for Treatment and Utilization of Municipal and Industrial Wastes. Proceedings of the Forest Lands Applications Symposium, Cole, D.W., C. Henry, and W.L. Nutter (eds.). University of Washington Press. Seattle, WA., Seattle, WA.

Correa, R.S., R.E. White, and A.J. Weatherley. 2005. Modelling the risk of nitrate leaching from two soils amended with five different biosolids. Revista Brasileira De Ciencia Do Solo 29(4):619-626.

Darrah, P.R., P.H. Nye, and W. R.E. 1987. The effect of high solute concentration on nitrifiction in soil. Plant and Soil 97:37-45.

Donohue, S.J., and S.E. Heckendorn. 1994. Soil test recommendations for Virginia. Virginia Tech. Blacksburg, VA.

Donovan, W.C., and T.J. Logan. 1983. Factors affecting ammonia volatilization from sewage-sludge applied to soil in a laboratory study. Journal of Environmental Quality 12(4):584-590.

Edmonds, R.L., and K.P. Mayer. 1981. Survival of sludge-associated pathogens and their movement into groundwater. P. 79-86 in Municipal sludge applicarion to Pacific Northwest forest land, Bledsoe, C.S. (ed.). Institute of Forest Resource, University of Washington, Seattle, WA.

Egiarte, G., M.C. Arbestain, A. Alonsoa, E. Ruiz-Romera, and M. Pinto. 2005. Effect of repeated applications of sewage sludge on the fate of N in soils under Monterey pine stands. Forest Ecology and Management 216(1-3):257-269.

Eick, M.J., W.D. Brady, and C.K. Lynch. 1999. Charge properties and nitrate adsorption of some acid southeastern soils. Journal of Environmental Quality 28(1):138-144.

Eldridge, S.M., K.Y. Chan, Z.H. Xu, C.R. Chen, and I. Barchia. 2008. Plant-available nitrogen supply from granulated biosolids: implications for land application guidelines. Australian Journal of Soil Research 46(5):423-436.

Epstein, E., D.B. Keane, J.J. Meisinger, and J.O. Legg. 1978. Mineralization of nitrogen from sewage sludge and sludge compost. Journal of Environmental Quality 7(2):217-221.

Evanylo, G.K. 1999a. Agricultural Land Application of Biosolids in Virginia: Managing Biosolids for Agricultural Use. Crop and Soil Environmental Sciences Publication. 452-303. Virginia Cooperative Extension Service. Blacksburg, VA.

Evanylo, G.K. 1999b. Agricultural Land Application of Biosolids in Virginia: Production and Characteristics of Biosolids. Crop and Soil Environmental Sciences Publication. 452-301 Virginia Cooperative Extension Service. Blacksburg, VA.

Evanylo, G.K. 1999c. Agricultural Land Application of Biosolids in Virginia: Risks and Concerns. Crop and Environmental Sicences Publications. 452-304. Virginia Cooperative Extension Service. Blacksburg, VA.

Evanylo, G.K. 2003. Effects of biosolids application timing and soil texture on nitrogen availability for corn. Communications in Soil Science and Plant Analysis 34(1-2):125-143.

Feigenbaum, S., A. Hadas, M. Sofer, and J.A.E. Molina. 1994. Clay-fixed labeled ammonium as a source of available nitrogen. Soil Science Society of America Journal 58:980-985.

Field, J.B. 1989. Water movement and chemical transport in a loblolly pine forest. *in* Georgia water resource conference, Kathryn, J. (ed.), University of Georgia, Athens, GA.

Fiskell, J.G.A., D.G. Neary, and N.B. Comerford. 1990. Slash Pine and Understory Interception of Micronutrients Mineralized from Sewage-Sludge Applied to a Sandy, Acidic Forest Soil. Forest Ecology and Management 37(1-3):27-36.

Fisher, R.F., and D. Binkley. 1999. Ecology and management of forest soils. 3rd edition. John Wiley & sons. 489 pp.

Flint, C.M., R.B. Harrison, B.D. Strahm, and A.B. Adams. 2008. Nitrogen leaching from douglas-fir forests after urea fertilization. Journal of Environmental Quality 37(5):1781-1788.

Forsbeg, C. 1998. Which policies can stop large scale eutrophication? Water Science Technology 37(3):193-200.

Fox, T.R., E.J. Jokela, and H.L. Allen. 2007a. The development of pine plantation silviculture in the southern United States. Journal of Forestry 105(7):337-347.

Fox, T.R., H.L. Allen, T.J. Albaugh, R. Rubilar, and C.A. Carlson. 2007. Tree nutrition and forest fertilization of pine plantations in the southern United States. Southern Journal of Applied Forestry 31(1):5-11.

Frank, J., and A.O. Stuanes. 2003. Short-term effects of liming and vitality fertilization on forest soil and nutrient leaching in a Scots pine ecosystem in Norway. Forest Ecology and Management 176:371-386.

Franklin, D.H., L.T. West, D.E. Radcliffe, and P.F. Hendrix. 2007. Characteristics and genesis of preferential flow paths in a Piedmont Ultisol. Soil Science Society of America Journal 71:752-758.

Gale, E.S., D.M. Sullivan, C.G. Cogger, A.I. Bary, D.D. Hemphill, and E.A. Myhre. 2006. Estimating plant-available nitrogen release from manures, composts, and specialty products. Journal of Environmental Quality 35(6):2321-2332.

Garau, M.A., M.T. Felipo, and M.C.R. Devilla. 1986. Nitrogen Mineralization of Sewage Sludges in Soils. Journal of Environmental Quality 15(3):225-228.

Gilmour, J.T. 1984. The effect of soil properties on nitrification and nitrification inhibition. Soil Science Society of America Journal 48(6):1262-1266.

Gilmour, J.T., M.D. Clark, and S.M. Daniel. 1996. Predicting long-term decomposition of biosolids with a seven-day test. Journal of Environmental Quality 25(4):766-770.

Gilmour, J.T., C.G. Cogger, L.W. Jacobs, G.K. Evanylo, and D.M. Sullivan. 2003. Decomposition and plant-available nitrogen in biosolids: Laboratory studies, field studies, and computer simulation. Journal of Environmental Quality 32(4):1498-1507.

Gilmour, J.T., Craig G. Cogger, Lee W. Jacobs, Gregory K. Evanylo, and Dan M. Sullivan. 2003. Decomposition and Plant-Available Nitrogen in Biosolids: Laboratory Studies, Field Studies, and Computer Simulation. Journal of Environmental Quality 32:1498-1507.

Gilmour, J.T., and V. Skinner. 1999. Predicting plant available nitrogen in land-applied biosolids. Journal of Environmental Quality 28(4):1122-1126.

Goldstein, N. 2007. Biosolids management trends in the U.S. BioCycle: Journal of Composting & Organic Recycling 48:9.

Gove, L., F.A. Nicholson, H.F. Cook, and A.J. Beck. 2002. Comparison of the effect of surface application and subsurface incorporation of enhanced treated biosolids on the leaching of heavy metals and nutrients through sand and sandy loam soils. Environmental Technology 23(2):189-198.

Grey, M., and Chuck Henry. 2002. Phosphorus and Nitrogen Runoff from a Forested Watershed Fertilized with Biosolids. Journal environmental quality 31:926-936.

Gurlevik, N., D.L. Kelting, and H.L. Allen. 2004. Nitrogen mineralization following vegetation control and fertilization in a 14-year-old loblolly pine plantation. Soil Science Society of America Journal 68(1):272-281.

Haith, D.A., J.E. Reynolds, P.T. Landre, and T.L. Richard. 1992. Sludge loading rates for forest land. Journal of Environmental Engineering-Asce 118(2):196-208.

Hallett, R.A., W.B. Bowden, and C.T. Smith. 1999. Nitrogen dynamics in forest soils after municipal sludge additions. Water Air and Soil Pollution 112(3-4):259-278.

Harrison, R., D.S. Xue, C. Henry, and D.W. Cole. 1994. Long-Term effects of heavy application of biosolids on organic-matter and nutrient content of a coarse-textured forest soil. P. 165-177 in IEA/BE Workshop on Ameliorative Practices for Restoring and Maintaining Long-Term Productivity in Forests, Vaxjo, Sweden.

Harrison, R.B., C.L. Henry, D.W. Cole, and D. Xue. 1995. Long-term changes in organic matter in soil receiving application of municipal biosolids. P. 139-153 in 8th North American Forest Soils Conference, McFee, W.W., J.M. Kelly, and J.M. Bigham (eds.), Gainsville, Fl.

Harrison, R.B., S.P. Gessel, D. Zabowski, C.L. Henry, D.S. Xue, D.W. Cole, and J.E. Compton. 1994b. Mechanisms of negative impacts of three forest treatments on nutrient availability. P. 1622-1628 in Symposium on Soil and Sustained Forest Productivity, at the Annual Meeting of the ASA-CSSA-SSSA, Seattle, Wa.

Hart, J.B., P.V. Nguyen, D.H. Urie, and D.G. Brockway. 1988. Silvicultural use of waste-water sludge. Journal of Forestry 86(8):17-24.

Hartenstein, R. 1981. Sludge decomposition and stabilization. Science 212(4496):743-749.

Hattori, H., and S. Mukai. 1986. Decomposition of sewage sludges in soil as affected by their organic matter composition. Soil science and plant nutrition 32:421-432.

Henry, C.L., D.W. Cole, and R.B. Harrison. 1994. Use of municipal sludge to restore and improve site productivity in forestry - The pack forest sludge research program. P. 137-149 in IEA/BE Workshop on Ameliorative Practices for Restoring and Maintaining Long-Term Productivity in Forests, Vaxjo, Sweden.

Henry, C.L., and D.W. Cole. 1997. Use of biosolids in the forest: Technology, economics and regulations. Biomass & Bioenergy 13(4-5):269-277.

Henry, C.L., D.W. Cole, T.M. Hinckley, and R.B. Harrison. 1993. The use of municipal and pulp and paper sludges to increase production in forestry. Journal of Sustainable Forestry 1(3):41-55.

Henry, C., D. Sullivan, R. Rynk, K. Dorsey, and C. Cogger. 1999. Managing nitrogen from biosolids. Washington State Dept. of Ecology, [Olympia? Wash.]. 75 p

Hernandez, T., R. Moral, A. Perez-Espinosa, J. Moreno-Caselles, M.D. Perez-Murcia, and C. Garcia. 2002. Nitrogen mineralisation potential in calcareous soils amended with sewage sludge. Bioresource Technology 83(3):213-219.

Hingston, F.J. 1981. A review of anion adsorption. in Adsorption of inorganics at solid–liquid interfaces, Anderson, M.A., and A.J. Rubin (eds.). Ann Arbor Sciences, Ann Arbor, MI.

Howarth, R.W. 1988. Nutrient limitation of net primary production in marine ecosystems. Ann. Rev. Ecol. Sys, 19:898-1110.

Iseman, T.M., D.R. Zak, W.E. Holmes, and A.G. Merrill. 1999. Revegetation and nitrate leaching from lake states northern hardwood forests following harvest. Soil Science Society of America Journal 63:1424-1429.

Jaynes, W.F., R.E. Zartman, R.E. Sosebee, and D.B. Wester. 2003. Biosolids decomposition after surface applications in west Texas. Journal of Environmental Quality 32(5):1773-1781.

Johnson, D.W., and D.W. Cole. 1980. Anion mobility in soils: Relevance to nutrient transport from terrestrial ecosystems. Environ. Int. 3:79–90.

Johnson, D.W., D.E. Todd, and V.R. Tolbert. 2003. Changes in ecosystem carbon and nitrogen in a loblolly pine plantation over the first 18 years. Soil Science Society of America Journal 67:1594-1601.

Jokela, E.J., W.H. Smith, and S.R. Colbert. 1990. Growth and elemental content of slash pine 16 years after treatment with garbage composted with sewage-sludge. Journal of Environmental Quality 19(1):146-150.

Jones, J.B., and W.J.A. Steyn. 1973. Sampling, handling, and analyzing plant tissue samples. pp. 249-270. Soil Sci. Soc. Amer., Madison, WI.

Jorgensen, J.R., C.G. Wells, and L.J. Metz. 1980. Nutrient Changes in Decomposing Loblolly-Pine Forest Floor. Soil Science Society of America Journal 44(6):1307-1314.

Jussy, J.H., M. Colin-Belgrand, E. Dambrine, J. Ranger, B. Zeller, and S. Bienaime. 2004. N deposition, N transformation and N leaching in acid forest soils. Biogeochemistry 69(2):241-262.

Kane, P.F. 2000. AOAC Methods. Association of Official Analytical Chemists.Washington, DC.

Katterer, T., M. Reichstein, O. Andren, and A. Lomander. 1998. Temperature dependence of organic matter decomposition: a critical review using literature data analyzed with different models. Biology and Fertility of Soils 27(3):258-262.

Kaufman, S.S., and D.A. Haith. 1986. Probabilistic Analysis of Sludge Land Application. Journal of Environmental Engineering-Asce 112(6):1041-1053.

Kelty, M.J., F.D. Menalled, and M.M. Carlton. 2004. Nitrogen dynamics and red pine growth following application of pelletized biosolids in Massachusetts, USA. Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 34(7):1477-1487.

Kinjo, T., and P.F. Pratt. 1971. Nitrate adsorption: I. in some acid soils of Mexico and South America. Soil Sci. Soc. Am. Proc. 35:722–725.

Kowalenko, C.G., and S. Yu. 1996. Assessment of nitrate adsorption in soils by extraction, equilibration and column-leaching methods. Canadian Journal of Soil Science 76(1):49-57.

Kroiss, H. 2003. What is the potential for utilizing the resources in sludge? P. 1-10 in International Conference on Wastewater Sludge as a Resource (BIOSOLIDS 2003), Trondheim, Norway.

Lal, R. 2003. Forest soils and carbon sequestration. P. 242-258 *in* 10th North American Forest Soils Conference, Saulte St Marie, Canada.

Leggett, Z.H., and D.L. Kelting. 2006. Fertilization effects on carbon pools in loblolly pine plantations on two upland sites. Soil Science Society of America Journal 70(1):279-286.

Leone, P. 2005. Review of Land Application of Biosolids in Virginia. P. 116, Commission, J.L.A.a.R. (ed.). Commonwealth of Virginia, Richmond, Virginia.

Lerch, R.N., K.A. Barbarick, L.E. Sommers, and D.G. Westfall. 1992. Sewage-Sludge Proteins as Labile Carbon and Nitrogen-Sources. Soil Science Society of America Journal 56(5):1470-1476.

Littell, R., G. Milliken, W. Stroup, R. Wolfinger, and O. Schabenberger. 2006. SAS for Mixed Models, Second Edition. SAS Press, Cary, NC.

Little, D.A., R.B. Reneau, and D.C. Martens. 1991. Lime-stabilized and chemical-fixed sewage sludges as lime amendments. Bioresource Technology 37(1):93-102.

Luo, Y.M., and P. Christie. 2001. Short-term effects of alkaline biosolids on pH and trace metals in oligotrophic forest peat and on growth of Picea sitchensis. Forestry 74(2):145-159.

Lowrance, R., R. Leonard, and J. Sheridan. 1985. Managing riparian ecosystems to control nonpoint pollution. J. Soil and Water Conservation 40(1):88-91.

Lowrance, R.R., T.J.F. Jr., O.H. Jr, R. Leonard, and L. Asmussen. 1984. Riparian forests as nutrient filters in agricultural watersheds. BioScience 34:374-377.

Luxmoore, R.J., M.L. Tharp, and R.A. Efroymson. 1999. Comparison of simulated forest responses to biosolids applications. Journal of Environmental Quality 28(6):1996-2007.

Lyngstad, I. 1992. Effect of Liming on Mineralization of Soil-Nitrogen as Measured by Plant Uptake and Nitrogen Released during Incubation. Plant and Soil 144(2):247-253.

Magesan, G.N., C.D.A. McLay, and V.V. Lal. 1998. Nitrate leaching from a free-draining volcanic soil irrigated with municipal sewage effluent in New Zealand. Agriculture Ecosystems & Environment 70(2-3):181-187.

Magill, A.H., J.D. Aber, G.M. Berntson, W.H. McDowell, K.J. Nadelhoffer, J.M. Melillo, and P. Steudler. 2000. Long-term nitrogen additions and nitrogen saturation in two temperate forests. Ecosystems 3(3):238-253.

Maguire, R.O., J.T. Sims, S.K. Dentel, F.J. Coale, and J.T. Mah. 2001. Relationships between biosolids treatment process and soil phosphorus availability. Journal of Environmental Quality 30(3):1023-1033.

Maimone, R.A., L.A. Morris, and T.R. Fox. 1991. Soil-Nitrogen mineralization potential in a fertilized loblolly-pine plantation. Soil Science Society of America Journal 55(2):522-527.

Malik, A., and J. Scullion. 1998. Soil development on restored opencase soil sites with particular reference to organic matter and aggregate stability. Soil use manage 14:234-238.

Matsuoka, K., N. Moritsuka, T. Masunaga, K. Matsui, and T. Wakatsuki. 2006. Effect of heating treatments on nitrogen mineralization from sewage sludge. Soil Science and Plant Nutrition 52(4):519-527.

McClung, G., and W.T. Frankenberger. 1985. Soil nitrogen transformatins as affected by salinity. Soil Sci 139:404-411.

McKee, W.H., Jr., K.W. McLeod, C.E. Davis, M.R. McKevlin, and H.A. Thomas. 1986. Growth response of loblolly pine to municipal and industrial sewage sludge applied at four ages on upper coastal plain sites. P. 272-281.

McLaren, R.G., L.M. Clucas, T.W. Speir, and A.P.v. Schaik. 2007. Distribution and movement of nutrients and metals in a Pinus radiata forest soil following applications of biosolids. Environmental Pollution 147(1):32-40.

McLaren, R.G., L.M. Clucas, and M.D. Taylor. 2005. Leaching of macronutrients and metals from undisturbed soils treated with metal-spiked sewage sludge. 3. Distribution of residual metals. Australian Journal of Soil Research 43(2):159-170.

McLaren, R.G., L.M. Clucas, M.D. Taylor, and T. Hendry. 2003. Leaching of macronutrients and metals from undisturbed soils treated with metal-spiked sewage sludge. 1. Leaching of macronutrients. Australian Journal of Soil Research 41(3):571-588.

Medalie, L., W.B. Bowden, and C.T. Smith. 1994. Nutrient leaching following land application of aerobically digested municipal sewage sludge in a northern hardwood forest. Journal of Environmental Quality 23(1):130-138.

Mehlich, A. 1953. Determination of P, Ca, Mg, K, Na, NH4. North Carolina Dept. of Agriculture, Agronomic Division.

Mehlich, A. 1984. Mehlich-3 soil test extractant - a modification of Mehlich-2 extractant. Communications in Soil Science and Plant Analysis 15(12):1409-1416.

Mitchell, D.S., A.C. Edwards, and R.C. Ferrier. 2000. Changes in fluxes of N and P in water draining a stand of Scots pine treated with sewage sludge. Forest Ecology and Management 139(1-3):203-213.

Moffet, C.A., R.E. Zartman, D.B. Wester, and R.E. Sosebee. 2005. Surface biosolids application: effects on infiltration, erosion, and soil organic carbon in Chihuahuan Desert grasslands and shrublands. J Environ Qual 34(1):299-311.

Mudano, J.E. 1986. Assessment of Soil Nitrogen Availability Following Nitrogen and Phosphorus Fertilization of a Loblolly Pine Stand, North Carolina State University, Raleigh, NC. 52 p.

Mullins, G.L., W.G. Alley, and S.B. Phillips. 2005. Sources of lime for acid soils in Virginia. 452-510. Virginia Cooperative Extention.

N.R.C. 2002. Biosolids applied to land : advancing standards and practices. National Academies Press. . xviii, 345 p.

Nguyen, P.V., J.B. Hart, Jr., and D.M. Merkel. 1986. Municipal sludge fertilization on oak forests in Michigan: short term nutrient changes and growth responses. P 282-291 in The forest alternative for tWastewater and sludge nutreatment and utilization of municipal and industrial wastes, D.W., C., C. Henry, and N. W.L. (eds.). University of Washington Press, Seattle.WA.

O'Connor, G.A., H.A. Elliott, N.T. Basta, R.K. Bastian, G.M. Pierzynski, R.C. Sims, and J.E. Smith. 2005. Sustainable land application: An overview. Conference on Sustainable Land Application:7-17.

Ollinger, S.V., M.L. Smith, M.E. Martin, R.A. Hallett, C.L. Goodale, and J.D. Aber. 2002. Regional variation in foliar chemistry and N cycling among forests of diverse history and composition. Ecology 83(2):339-355.

Parnaudeau, V., B. Nicolardot, and J. Pages. 2004. Relevance of organic matter fractions as predictors of wastewater sludge mineralization in soil. Journal of Environmental Quality 33(5):1885-1894.

Parnaudeau, V., B. Nicolardot, P. Robert, G. Alavoine, J. Pages, and F. Duchiron. 2006. Organic matter characteristics of food processing industry wastewaters affecting their C and N mineralization in soil incubation. Bioresource Technology 97(11):1284-1295.

Penn, C.J., and J.T. Sims. 2002. Phosphorus forms in biosolids-amended soils and losses in runoff: Effects of wastewater treatment process. Journal of Environmental Quality 31(4):1349-1361.

Pepper, I.L., H. Zerzghi, J.P. Brooks, and C.P. Gerba. 2008. Sustainability of land application of class B biosolids. Journal of Environmental Quality 37:58-67.

Pocknee, S., and M.E. Sumner. 1997. Cation and nitrogen contents of organic matter determine its soil liming potential. Soil Science Society of America Journal 61(1):86-92.

Powers, R.F. 1980. Mineralizable soil-nitrogen as an index of nitrogen availability to forest trees. Soil Science Society of America Journal 44(6):1314-1320.

Prescott, C.E., and L.L. Blevins. 2005. Eleven-year growth response of young conifers to biosolids or nitrogen and phosphorus fertilizer on northern Vancouver Island. Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 35(1):211-214.

Prescott, C.E., M.A. McDonald, S.P. Gessel, and J.P. Kimmins. 1993. Long-term effects of sewage-sludge and inorganic fertilizers on nutrient turnover in litter in a coastal Douglas-fir forest. Forest Ecology and Management 59(1-2):149-164.

Prescott, C.E., and S.M. Brown. 1998. Five-year growth response of western red cedar, western hemlock, and amabilis fir to chemical and organic fertilizers. Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 28(9):1328-1334.

Pritchett, W.L., and R.F. Fisher. 1987. Properties and management of forest soils. John Wiley, New York, NY.494 pp.

Quemada, M., B. Lassa, C. Lamsfus, and P.M. Aparicio Tejo. 1998. Ammonia Volatilization from Surface or Incorporated Biosolids by the Addition of Dicyandiamide. Journal of Environmental Quality 27:980-983.

Raison, R.J., M.J. Connell, and P.K. Khanna. 1987. Methodology for Studying Fluxes of Soil Mineral-N Insitu. Soil Biology & Biochemistry 19(5):521-530.

Raison, R.J., M.J. Connell, P.K. Khanna, and R.A. Falkiner. 1992. Effects of Irrigation and Nitrogen-Fertilization on Fluxes of Soil Mineral Nitrogen in a Stand of Pinus-Radiata. Forest Ecology and Management 52(1-4):43-64.

Richter, D.D., D. Markewitz, J.K. Dunsomb, P.R. Heine, C.G. Wells, A.O. Stuanes, H.L. Allen, B. Urrego, K. Harrison, and G. Bonani. 1995. Carbon cycling in a loblolly pine forest: Implication for the missing carbon sink and fore the concept of soil. in Carbon forms and function in forest soil, McFee, W.W., and J.M. Kelly (eds.). Soil science society of America Inc, Gainsville, Florida.

Riekerk, H. 1978. The behavior of nutrient elements added to a forest soil with sewage sludge. Soil Science Society of America Journal 42(5):810-816.

Riekerk, H. 1981. Effects of sludge disposal on drainage solutions of 2 forest soils. Forest Science 27(4):792-800.

Robinson, M.B., and P.J. Polglase. 2000. Volatilization of nitrogen from dewatered biosolids. Journal of Environmental Quality 29(4):1351-1355.

Robinson, M.B., P.J. Polglase, and C.J. Weston. 2002. Loss of mass and nitrogen from biosolids applied to a pine plantation. Australian Journal of Soil Research 40(6):1027-1039.

Robinson, M.B., and H. Roper. 2003. Volatilisation of nitrogen from land applied biosolids. Australian Journal of Soil Research 41(4):711-716.

Rostagno, C.M., and R.B. Sosebee. 2001. Surface application of biosolids in the Chihuahuan Desert: Effects on soil physical properties. Arid Land Research and Management 15(3):233-244.

Rowell, D.M., C.E. Prescott, and C.M. Preston. 2001. Decomposition and nitrogen mineralization from biosolids and other organic materials: Relationship with initial chemistry. Journal of Environmental Quality 30(4):1401-1410.

Schroder, J.L., H. Zhang, D. Zhou, N. Basta, W.R. Raun, M.E. Payton, and A. Zazulak. 2008. The effect of long-term annual application of biosolids on soil properties, phosphorus, and metals. Soil Science Society of America Journal 72(1):73-82.

Sidle, R.C., and L.T. Kardos. 1979. Nitrate leaching in a sludge-treated forest soil. Soil Science Society of America Journal 43(2):278-282.

Silva, R.G., S.M. Holub, E.E. Jorgensen, and A.N.M. Ashanuzzaman. 2005. Indicators of nitrate leaching loss under different land use of clayey and sandy soils in southeastern Oklahoma. Agriculture Ecosystems & Environment 109(3-4):346-359.

Singh, R.P., and M. Agrawal. 2007. Potential benefits and risks of land application of sewage sludge. P. 347-358 *in* 30th Annual Meeting of the Society-of-General-Internal-Medicine, Toronto, Canada.

Simmons, J.A., J.B. Yavitt, and T.J. Fahey. 1996. Watershed liming effects on the forest floor N cycle. Biogeochemistry 32(3):221-244.

Smith, M.T.E., R.J. Smernik, G. Merrington, and M. Tibbett. 2008. Changes in sewage sludge carbon forms along a treatment stream. Chemosphere 72(6):981-985.

Smith, S.R., and E. Durham. 2002. Nitrogen release and fertiliser value of thermally-dried biosolids. Journal of the Chartered Institution of Water and Environmental Management 16(2):121-126.

Smith, S.R., V. Woods, and T.D. Evans. 1998. Nitrate dynamics in biosolids-treated soils. I. Influence of biosolids type and soil type. Bioresource Technology 66:139-149.

Smith, V.H., G.D. Tilman, and J.C. Nekola. 1999. Eutrophication: impacts of excess nutrient imouts on freshwater, marine, and terrestrial ecosystems. . Environmental Pollution 100:179-196.

Sollins, P., G.P. Robertson, and G. Uehara. 1988. Nutrient mobility in variable and permanent-charge soils. Biogeochemistry 6:181-199.

Sommers, L.E. 1977. Chemical composition of sewage sludges and analysis of their potential use as fertilizers. Journal of Environmental Quality 6(2):225-232.

Sommers, L.E., D.W. Nelson, and D.J. Silviera. 1979. Transformations of carbon, nitrogen, and metals in soils treated with waste materials. Journal of Environmental Quality 8(3):287-294.

Strahm, B.D., and R.B. Harrison. 2006. Nitrate sorption in a variable-charge forest soil of the Pacific Northwest. Soil Science 171(4):313-321.

Strahm, B.D., and R.B. Harrison. 2007. Mineral and organic matter controls on the sorption of macronutrient anions in variable-charge soils. Soil Science Society of America Journal 71(6):1926-1933.

Strahm, B.D., and R.B. Harrison. 2008. Controls on the Sorption, Desorption, and Mineralization of Low-Molecular-Weight Organic Acids in Variable-Charge Soils. Soil Science Society of America Journal 72(6):1653-1664.

Subler, S., J.M. Blair, and C.A. Edwards. 1995. Using Anion-Exchange Membranes to Measure Soil Nitrate Availability and Net Nitrification. Soil Biology & Biochemistry 27(7):911-917.

Swanston, C., P.S. Homann, B.A. Caldwell, D.D. Myrold, L. Ganio, and P. Sollins. 2004. Long-term effects of elevated nitrogen on forest soil organic matter stability. Biogeochemistry 70(2):227-250.

Tarrason, D., G. Ojeda, O. Ortiz, and J.M. Alcaniz. 2008. Differences on nitrogen availability in a soil amended with fresh, composted and thermally-dried sewage sludge. Bioresource Technology 99(2):252-259.

Terry, R.E., D.W. Nelson, and L.E. Sommers. 1979. Decomposition of anaerobically digested sewage sludge as affected by soil environmental-conditions. Journal of Environmental Quality 8(3):342-347.

Terry, R.E., D.W. Nelson, and L.E. Sommers. 1981. Nitrogen transformation in sewage sludge amended soils as affected by soil environmental factors. Soil Science Society of America Journal 45(3):506-513.

Tisdale, S.L., W.E. Nelson, and J.D. Beaton. 1985. Soil fertility and Fertilizer.4th edition. Mcmillan. New York, NY. 754 pp.

Toner, C.V.I., D.L. Sparks., and T.H. Carski. 1989. Anion Exchange Chemistry of Middle Atlantic Soils: Change Properties and Nitrate Retention Kinetics. Soil Science Society of America Journal 53:1061-1067.

U.V.A. 1997. Land application of biosolids in Virginia: A Study prepared for the Virginia Department of Health. UVA Institute for Environmental Negotiation. 47.

USEPA. 1983. Methods for the chemical analysis of water and wastes (MCAWW). EPA/600/4-79/020. NTIS item PB84-128677. in Environ. Monitoring and Support Lab. Office of Res. and Dev., USEPA, Cincinnati, OH.

USEPA. 1986. Quality criteria for water EPA-440/5-86-001. U.S. Environmental protection agency.Washington, DC.

USEPA. 1994. Biosolids recycling: beneficial technology for a better environment. EPA-832/R-94-009. U.S. Environmental protection agency. Washington, DC. 25 pp.

USEPA. 1995. Process design for agricultural utilization. In Process Design Manual–Land Application of Sewage Sludge and Domestic Septage EPA/625/R-95/001; U.S. Environmental protection agency. Washington, DC.

USEPA. 2000. Biosolids Technology fact sheet. Land application of biosolids. EPA 832-F-00-052. Office of water. U.S. Environmental protection agency. Washington, DC.9 pp.

USEPA. 2002. Test methods for evualating solid waste, physical/chemical methods SW-846 manual U.S. Environmental protection Agency. U.S. Gov Print Office.

Van Lear, D.H., and P.R. Kapeluck. 1995. Distribution of carbon in a Piedmont Soil as affected by Loblolly pine Management. in Carbon forms and functions in forest soils, McFee, W.W., and J.M. Kelly (eds.). Soil Society of America, Inc, Madison, Wisconsin, USA.

VDH. 2004. Report of the Virginia Department of Health on the U.S. Environmental Protection Agency's response to the National Research Council's report pertaining to the land application of biosolids to the Governor and the General Assembly of Virginia. Commonwealth of Virginia, Richmond, Va. 20 p. p.

Vitousek, P.M., S.W. Andariese, P.A. Matson, L. Morris, and R.L. Sanford. 1992. Effects of harvest intensity, site preparation, and herbicide use on soil nitrogen transformations in a young loblolly pine plantation. Forest Ecology and Management 49(3-4):277-292.

Vitousek, P.M., K.v. Cleve, N. Balakrishnan, and D. Mueller-Dombois. 1983. Soil development and nitrogen turnover in montane rainforest soils on Hawai'i. Biotropica 15(3):268-274.

Vitousek, P.M., and P.A. Matson. 1984. Mechanisms of nitrogen retention in forest ecosystems: A field experiment. Science 225:51-52.

Vitousek, P.M., and P.A. Matson. 1985. Disturbance, nitrogen availability, and nitrogen losses in a intensively managed loblolly pine plantation. Ecology 66:1360-1376.

Wang, H.L., M.O. Kimberley, and M. Schlegelmilch. 2003. Biosolids-derived nitrogen mineralization and transformation in forest soils. Journal of Environmental Quality 32(5):1851-1856.

Wang, H.L., G.N. Magesan, M.O. Kimberley, T.W. Payn, P.J. Wilks, and C.R. Fisher. 2004. Environmental and nutritional responses of a Pinus radiata plantation to biosolids application. Plant and Soil 267(1-2):255-262.

Wang, H., G.N. Magesan, P.W. Clinton, and J.M. Lavery. 2004b. Using natural N-15 abundances to trace the fate of waste-derived nitrogen in forest ecosystems: New Zealand case studies. P. 31-38 in 4th International Conference on Applications of Stable Isotope Techniques to Ecological Studies, Wellington, New Zealand.

Wang, H., M.O. Kimberley, G.N. Magesan, R.B. McKinley, J.R. Lee, J.M. Lavery, P.D.F. Hodgkiss, T.W. Payn, P.J. Wilks, C.R. Fisher, and D.L. McConchie. 2006. Midrotation effects of biosolids application on tree growth and wood properties in a Pinus radiata plantation. Canadian Journal of Forest Research-Revue Canadienne De Recherche Forestiere 36(8):1921-1930.

Wells, C.G., C.E. Murphy, C. Davis, D.M. Stone, and G.J. Hollod. 1986. Effect of sewage sludge from two sources on element flux in soil solution of loblolly pine plantations. P. 154-165

in The Forest Alternative for Treatment and Utilization of Municipal and Industrial Wastes, Cole, D.W., C. Henry, and W.L. Nutter (eds.). University of Washington Press, Seattle and London.