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CARBON AND NITROGEN DYNAMICS IN COAL MINE SOILS RECLAIMED WITH POULTRY MANURE

A Thesis in

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by

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ABSTRACT

In Pennsylvania, 150 years of extensive coal mining has left an estimated 250,000 acres of severely degraded mined land and impaired streams. In the same region, concentrated animal production facilities produce manure in excess of crop needs, creating an increased risk of nutrient pollution in surface and groundwater. Excess poultry manure could be used in mine reclamation, but the large application rates required for successful revegetation could result in significant nutrient discharge. Furthermore, abandoned mine lands could be used to grow switchgrass (*Panicum virgatum* L.), a biofuel crop, to return these lands to productivity. To achieve high production rates, however, significant and sustained nutrient levels are necessary. The goal of this research is to identify how poultry manure could be used on abandoned mine lands to ameliorate soil phytotoxicity, minimize nutrient losses, sequester nutrients and produce high switchgrass yields. Greenhouse, lab and field studies were designed to test two different methods of using poultry manure, composting and C:N ratio adjustment, to achieve this goal.

In a greenhouse experiment, columns of mine soil were amended with fresh manure, manure mixed with short fiber paper mill sludge (C:N ratios of 20 - 40) and 3 rates of composted manure and leached every four weeks to assess leaching loss of macronutrients and switchgrass growth. Fresh manure exhibited the highest leaching of $NO_3^- – N$ (192 mg column^{-1}); increasing the C:N ratio to 30:1 resulted in a five-fold decrease in the amount of NO_3^- leached. All compost treatments leached less than 6% of added N despite large application rates. Less than 2% of added P was leached from all treatments, indicating no significant risk of P leaching loss. There was a linear increase in switchgrass growth with each level of compost addition fresh with paper mill sludge. These results confirmed the idea that poultry manure N losses could be minimized by C:N adjustment and produce more switchgrass than lime and fertilizer amended soils.

A lab incubation experiment maximized N mineralization to determine stable and labile N and C pools associated with various treatments, which included lime plus fertilizer, two rates of composted poultry manure and two blends of fresh poultry layer manure mixed with paper mill sludge with C:N ratios of 20:1 and 30:1. These treatments were incubated immediately following amendment application (year one) and one year after field application (year two). In both incubations, organic treatments were more effective at building large stable N and C pools compared to lime plus fertilizer, confirming that both methods of C:N adjustment are effective at retaining N and C and are significantly greater than N and C pools associated with inorganic fertilizer. Manure and paper mill sludge, however, had more microbial biomass after two years, which could translate to enhanced, long-term nutrient cycling. There was no measured benefit of additional carbon added with the 30:1 C:N adjustment.

A field experiment using the same treatments as the lab incubation confirmed that both C:N adjustment methods work equally well to revegetate the mine soil and eliminate phytotoxic conditions. Leaching losses from composts were <1% of added N, while manure and paper mill sludge lost 8% and 16% of added N with a 20:1 and 30:1 C:N ratio, respectively. Organic treatments sequestered greater C and N in soil compared to lime plus fertilizer. Although revegetation was successful for all treatments, manure and paper mill sludge treatments produced superior switchgrass yields after three years.

Both composting poultry manure and mixing paper mill sludge with fresh poultry layer manure can be used to effectively minimize phytotoxic mine soil conditions, minimize N leaching losses from poultry manure, and produce high switchgrass yields. Both of these treatments also generated larger stable N and C pools, implying greater sustainability of nutrient cycling. Furthermore, increased microbial activity and higher yields with fresh manure and paper mill sludge suggest increased benefits from direct application of these materials. This new approach could help alleviate the problems of both abandoned minelands and excess manure in Pennsylvania, while potentially generating revenue from switchgrass production.

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Chapter I

Introduction and Objectives

In many areas of Pennsylvania, coal production has severely degraded much of the land and has contributed acid mine drainage and eroded sediments to nearby waters, impairing water quality. In all, there are over 250,000 acres of unreclaimed surface mine lands in Pennsylvania alone (PADEP, 1996) (Fig. 1-1). The soil at abandoned mines often supports poor vegetative cover due to acidity, limited water holding capacity, degraded soil physical properties, and low levels of organic matter and nutrients, especially nitrogen (N) and phosphorus (P) (Bendfeldt et al., 2001). Acidic soil conditions produce Al^{3+} phytotoxicity, which severely limits plant growth in these soils (von Willert and Stehouwer, 2003). Soil disturbance associated with mining activities causes the loss of large quantities of soil organic carbon (SOC) by mineralization, erosion and leaching (Akala and Lal, 2001). Low organic C levels in mine spoils limit microbial activity at these sites, which severely inhibits restoration of soil nutrient cycling (Lachlan et al., 2005; Machulla et al., 2005). The generally low soil microbial biomass associated with these sites is likely also influenced by slightly toxic conditions for microbes, which can hinder microbial growth (Stroo and Jenks, 1982; Akala and Lal, 2001).

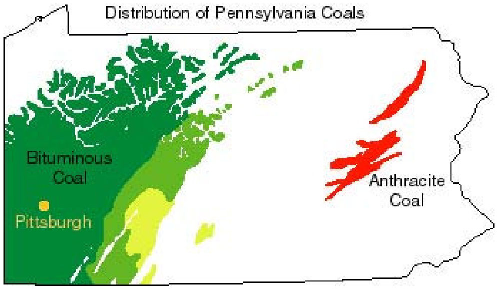

Figure 1-1. Coal regions of Pennsylvania.

While these mine sites are considered severely degraded and are not widely suitable for conventional agriculture, there is great potential to restore these lands to more normally functioning ecosystems. Furthermore, these barren lands have the potential to store C and other nutrients, such as N and P (Robertson et al., 2000). It has been suggested that reclaimed mine land in Pennsylvania alone could sequester 2.4 to 4.8 Tg C over 20 years if reclaimed to pasture (Sperow, 2006). Although this represents <1% of the 1990 CO_2 emissions for the region, potential sequestration in mine soils in the northeast could still account for 4 to 12.5% of reductions required by the Kyoto Protocol. Mine soils can potentially sequester 0.1 to 3.1 Mg C ha^{-1} yr^{-1} of soil organic C, which would also significantly contribute to enhanced soil functioning in addition to atmospheric C reductions (Shrestha and Lal, 2006; Akala and Lal).

Current Reclamation Practices

According to the Surface Mining Control and Reclamation Act of 1977 (SMCRA, Public Law 95-87), all mined land is required to be restored to a land use that is equal or better to pre-mining conditions. By far the most common reclamation practice, the addition of ground limestone and fertilizer followed by seeding with mixed grasses, is used to raise soil pH and add sufficient N, P and K to establish vegetation. Bloomfield et al. (1982) found that this one time application of fertilizer, however, did not sufficiently build up nutrients in the soil, leaving reclaimed land extremely deficient in nutrients, especially N. The lack of nutrient build up in soils would therefore require additional inputs to maintain plant productivity.

A more recent reclamation method is the direct application of sewage sludge to mine lands. Sewage sludge, also called biosolids, is the by-product of wastewater treatment facilities. This material contains large quantities of nutrients and numerous studies have shown improved reclamation of mine sites with use of this material compared to limestone and fertilizer, particularly when topsoil or overburden are low in organic matter (Hearing et al., 2000). While biosolids application to mine sites is an efficient way to use the waste material and improve reclamation success, there is an extremely high risk of nutrient loss due to leaching, especially when applied at the recommended rate of 134 Mg ha^{-1} (Stehouwer et al., 2006; Li and Daniels, 2001; Kostyanovskiy et al., 2008). In terms of water quality, especially in the sensitive Chesapeake Bay watershed, the environmental implications of applying biosolids do not appear to outweigh the benefits of improved vegetative growth on mine sites, implying better methods to using this material are necessary.

Reclamation economics

The economics of mine reclamation necessitate an inexpensive, one-time application of materials that will require no further input or care once mining activities have ceased. Given that materials are applied only once, benefits to soil and vegetation associated with these amendments must be contained within that material. Conversely, the limitation of applying material only once encourages large additions of material which increases the risk of nitrate leaching to groundwater; this effect, however, is assumed to be short-term (Hearing et al., 2000). Organic amendments, such as manure, have been shown to improve the fertility of the abandoned mine soil and restore a balance of nutrients if applied in proper ratios, making them an ideal candidate for a one time application in mine reclamation (Seaker and Sopper, 1988; Bendfeldt et al., 2001; Hearing et al., 2000). Furthermore, proper use can encourage the development of self-sustaining plant communities, one of the main goals in restoring these lands.

Poultry manure

In many areas of Pennsylvania, concentrated poultry operations produce manure in excess of local crop needs, resulting in increased transport of nitrogen and phosphorus to water bodies where they have been identified as the main pollutants damaging ecosystem and water quality (Kellog et al., 2000). Poultry manure in particular is a problem, as this industry is often concentrated on small parcels and lacks a land base for the manure it produces (Moore et al., 1995). In combination with other animal production in the region, poultry production causes an imbalance of nutrients in the region due to accumulation of excess manure, leaving much land enriched in P and N in the Chesapeake Bay watershed (Chesapeake Bay Program, 2005). There is a demand for manure on farmland in the Midwest, but transportation of this material is impractical both economically and logistically. It is necessary, therefore, to find a beneficial use for the manure nearby. Abandoned mine land would be a good candidate for receiving this excess material, as these acidic soils are severely degraded and lacking in organic matter and nutrients (Bendfeldt et al., 2001).

Manure has long been used in agricultural settings to add nutrients, especially N, to the soil and improve overall soil quality as compared to inorganic fertilizer (Edmeades, 2003). There is also evidence of long-term soil benefits of adding fresh manure or sewage sludge to soils as opposed to applying more stabilized materials, wherein properties such as soil organic matter and microbial biomass are measurably greater in soils amended with fresh material even after 8 yr (Pascual et al., 1999).

Application of large quantities of manure over time has been shown to cause excessively high nutrient levels in soil, especially P, which are at risk for leaching (Sharpley et al., 1996). Numerous studies have also documented increased N and P losses by leaching and runoff losses when fresh manure alone is applied to soil (Flavel and Murphy, 2006; Eghball and Gilley, 1999; Hanselman et al., 2004; Carpenter et al., 1998). Edmeades (2003) showed that large quantities of manure over many years were required to improve soil quality under field conditions and with this comes a large risk of nutrient leaching, especially of nitrogen. The low C:N ratio of manure (7:1), coupled with the lack of organic C in mine soils, necessitates large applications of manure to meet both N and organic C requirements of the soil, which would inevitably lead to large nutrient losses (Bendfeldt et al., 2001).

Biofuel production

Currently unproductive mine lands could potentially be used to produce a crop such as switchgrass (*Panicum virgatum* L.), which requires minimal fertilizer inputs and could make biofuel if harvested and pelletized (Fike et al., 2006). For this to be feasible, not only would phytotoxic mine soil conditions need to be eliminated, but adequate nutrients would be necessary to support high yields. Switchgrass could be harvested once per year in the fall, thereby generating biomass, minimizing N and other nutrient removal from the soil, and maintaining soil surface cover to prevent erosion (Reynolds et al., 2000; Sanderson et al., 1999). Furthermore, as biofuel production systems require low inputs to be economical, the nutrient retention of these soils must be such that frequent additions of nutrients are not necessary to maintain vegetative growth (Tilman et al., 2006).

Two Potential Solutions

Because excess poultry manure exists within the same region as abandoned strip mines, it is conceivable to use the former to improve the latter, effectively ameliorating both environmental problems. In order to use manure, however, potential environmental impacts must be minimized, especially in the Chesapeake Bay watershed, which suffers from excess nitrogen and phosphorous inputs (Chesapeake Bay Program, 2009; Carpenter et al., 1998). This research investigates two different approaches to using waste materials for mineland reclamation: composting poultry layer manure or adjusting the C:N ratio of raw poultry layer manure with the addition of paper mill sludge.

Composting poultry manure

Land application of composted organic materials is a common method of adding both nutrients and organic matter to the soil, and the use of compost to improve soil quality and minimize N loss by leaching has been well documented (Amlinger et al., 2003; Bernal et al., 1998; Gallardo-Lara and Nogales, 1987; Preusch et al., 2002; Lynch et al., 2004). In the process of composting, the combination of a high C:N ratio material, such as leaf and yard material, with a low C:N ratio material, such as manure, accelerates microbial transformation of labile organic compounds in the materials into more stable humus-like material. This process lowers the C:N ratio as CO_2 is released; as a result, readily available N is transformed into more stable compounds (Brady and Weil, 2008). The incorporation of N into complex, humus-like molecules prevents N from moving through the soil and into groundwater, making it easier to manage this highly mobile nutrient (Kluge, 2006; Amlinger et al., 2003). Compost provides a stable nutrient source for plant growth due to low mineralization rates of N, which builds a stable pool of N in the soil with time (Amlinger et al., 2003; Hartl and Erhart, 2005).

When poultry manure is properly composted, the potential environmentally harmful effects of ammonium and nitrate leaching can be greatly reduced or eliminated (Nahm, 2005). Studies have demonstrated that even with large compost application rates, quantities of nitrate in soil and leachate are low, especially compared to fresh poultry manure or inorganic fertilizer amended soils (Cooperband et al., 2002; Hartl et al., 2000). Furthermore, studies have shown improvement of soil properties, such as hydraulic conductivity, water retention capacity, bulk density, porosity, and aggregate stability, proportional to the rate of compost addition (Aggelides and Londra, 2000). Compost also adds significant quantities of stable organic matter to soils, increased cation exchange capacity, and soil pH with the addition of compost to soils (Eghball and Gilley, 1999; Aggelides and Londra, 2000; Gallardo-Lara and Nogales, 1987).

Although compost is extremely stable once applied to soil, approximately 46 to 62% of C and up to 40% of N, depending on initial C:N ratio of the materials, can be lost in the process of composting (Eghball et al., 1997). An additional concern with the use of compost is N immobilization in soil which leaves plants N starved despite large quantities of N in the soil (Douglas and Magdoff, 1991). The slow release of N from these materials is ideal for preventing N buildup in soils in excess of plant needs, but it can also be released too slowly, especially when N demand from plants is high.

Adjusting manure C:N ratio

Another approach would be to adjust the C:N ratio of fresh manure with a high C material, which has been shown to increase N immobilization, and thereby minimize N mineralization, as the C:N is increased (Vigil and Kissel, 1991). Many studies have shown improved soil ecosystem recovery and greater yields with the co-application of animal manures or biosolids with high carbon materials such as paper mill sludge (PMS) or sawdust compared to inorganic amendments (Haering et al., 2000; Bellamy et al., 1995; Schmidt et al., 2001).

Paper mill sludge is a good option to add to fresh poultry layer manure because it can help increase soil organic matter, improve soil physical properties, retain nutrients, and increase soil pH (Camberato et al., 2006; Fiero et al., 1997). Paper mill sludge is a by-product of the paper industry, of which there are many factories located in the Mid-Atlantic region. These plants produce roughly 40,000 tons of paper sludge per year, most of which is land filled (PADEP, 2009). As landfill costs rise, manufacturers are looking to find other uses for the waste product. Although some companies charge for the material, others still offer it free of charge excluding transportation costs (PADEP). The high carbon content of the mostly cellulosic short fibers in the paper mill sludge are ideal for adding carbon to the soil. Furthermore, the alkaline properties of PMS provide a liming effect on soils, thereby minimizing phytotoxic soil conditions associated with low pH and high metal mobility (Hearing et al., 2000).

Adding fresh manure to soil has also been shown to improve long-term soil microbial activity and it has also been suggested that less processed materials, such as sewage sludges and fresh manure, provide a better microbial food supply than composted material (Pascual et al., 1997; Gigliotti et al., 2002). This implies an *in situ* benefit of applying raw manure to the soil where the decomposition processes that would have occurred in the compost pile are happening directly in the soil. Additionally, the cost of transporting these materials to a mine site for reclamation is significantly cheaper; compost incurs additional costs in the manufacture and transport of the material, an important fact to consider when evaluating mine reclamation methods.

Minimal research has been conducted on the nitrogen dynamics of mine soil amended with fresh poultry manure and high carbon materials. Greenhouse studies have shown reduced N leaching with the co-application of poultry manure and paper mill sludge, and field studies using paper mill sludge and inorganic fertilizer have produced higher yields than with inorganic fertilizer alone (Busscher et al., 1999; Feagly et al., 1994). Daniels et al. (2001) demonstrated reduced nitrate leaching with co-application of sawdust and biosolids compared to the addition of biosolids alone. The higher mineralization rates associated with fresh manure, however, could still be of concern even when applied in conjunction with a high C material, and therefore must be tested.

Hypotheses and Objectives

Because the environmental problems of excess manure, paper by-products, and degraded mined lands exist within the same region, it is potentially feasible to combine the three to solve problems of excess waste and barren mine land. I hypothesize that:

1. The addition of a paper mill sludge to fresh poultry manure or composting poultry manure will retain more nutrients, and lose fewer nutrients to leaching, than the application of fresh manure alone,

2. The use of either composted poultry manure or fresh poultry layer manure mixed with paper mill sludge in coal mine reclamation will mitigate soil phytotoxicity, sequester nutrients, minimize nutrient leaching, and increase vegetative production to a greater extent than with traditional lime and fertilizer amendments,

3. The application of fresh manure and paper mill sludge to mine soils will be as effective at eliminating soil phytotoxicity, minimizing nutrient loss, and retaining nutrients, and will have greater switchgrass yields, compared to adding composted poultry manure to mine soils.

To test these hypotheses, I conducted three experiments investigating mine soil amelioration with poultry manure that was unprocessed, composted, or combined with paper mill sludge. The three experiments were conducted under several different environmental conditions. A short-term study in the greenhouse served as a proof of concept experiment while also examining specific rates of application under controlled environmental conditions. A lab incubation study served to test the N and C mineralization potential under a “worst case scenario” environment of intensive leaching, while also determining the stable N and C pools resulting from various manure amendments. A long-term field study was employed to test these amendment materials behave under real-world conditions and also to assess vegetative response to the different materials in a natural environment. Specific objectives for each experiment are listed below.

Greenhouse experiment

1. Measure the effects of composted poultry manure or fresh poultry manure mixed with paper mill sludge on nutrient leaching and soil phytotoxicity.
2. Determine appropriate application rates for minimizing nutrient loss, eliminating soil phytotoxicity and generating switchgrass growth.

Lab incubation experiment

1. Measure the extent of N leaching associated with composted or fresh poultry manure mixed with paper mill sludge by maximizing N mineralization.
2. Determine N and C sequestration into stable soil pools one and two years after application of composted or fresh manure mixed with paper mill sludge.

Field experiment

1. Measure the extent to which composting or adding paper mill sludge to poultry manure controls nutrient leaching when used for mine reclamation.
2. Measure the extent to which composting or adding paper mill sludge to poultry manure enhances soil sequestration of nitrogen and carbon.
3. Measure switchgrass productivity associated with composted or fresh manure with paper mill sludge treatments in reclaimed mine soil.

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Chapter II

Nutrient leaching and switchgrass growth in mine soil columns amended with poultry manure

Introduction

The low C:N ratio and large quantities of readily mineralizable N in poultry manure mean that manure N, although readily available for plant use, is highly susceptible to leaching losses (Flavel and Murphy, 2006). One approach to reducing N loss is the widen the C:N ratio by mixing manure with a high C material. This adjustment should stabilize N so that it is not so susceptible to leaching losses, yet is also available for plant use.

If a C:N ratio is too high, it is highly likely that N immobilization will occur. Parker and Sommers (1983), in an experiment analyzing the mineralization rates of biosolids in a silt loam soil, reported an inverse relationship between N mineralization and C:N ratio, with N immobilization occurring when the C:N ratio approached 25:1. Vigil and Kissel (1991) determined N immobilization occurred when C:N ratios of crop residues were greater than 40:1. By contrast, Douglas and Magdoff (1991), in an incubation study using fresh and composted manures and biosolids, found N immobilization occurring at C:N ratios less than 25:1. Clearly, the C:N ratio is an important factor dictating the ability of compost or manure and paper mill sludge to minimize N losses but also provide N for plant growth.

Similarly, the rate at which N will be mineralized is affected by the degree of stabilization of that material. N and C loss, as well as changes in soil microbial biomass, have all been noted to differ based on degree of stabilization of manures or biosolids: less stable materials lose more N and C, but also have more microbial activity, than stabilized materials (Bernal et al., 1998; Sanchez-Monedero et al., 2004; Pascual et al., 1997). Compost, in which manure N has been almost completely stabilized, would be expected to retain N effectively, but may not successfully reclaim mine lands if N is released too slowly and consequently not available for plant growth. Likewise, given that manure has not been stabilized prior to application, more N losses would be expected with application of raw manure (Eghball, 2000). Adding a high C material, and therefore adjusting the C:N ratio, would also be expected to minimize leaching losses, but it is unclear exactly how much this would reduce N loss or impact vegetative growth.

Paper mill sludge is an ideal candidate for co-application with fresh poultry layer manure because it can help increase soil organic matter, improve soil physical properties, retain nutrients, and increase soil pH (Camberato et al., 2006). In a greenhouse experiment performed by Feagly et al. (1994), paper mill sludge was added at different rates to mine soil with and without fertilizer. They found that the addition of paper mill sludge and fertilizer to mine soils significantly increased yields over soils amended only with fertilizer. Fiero et al. (1997) also determined that paper mill sludge is a beneficial soil amendment, but only when adequate nutrients are applied; if not applied in conjunction with an N source, N immobilization is likely.

Because greenhouse experiments are ideal for looking at treatments under conditions both more controlled and intensive than found in a natural environment, I assembled soil columns with an accelerated leaching schedule and frequent biomass harvests to test:

1. the effects of composted poultry manure or fresh poultry manure mixed with paper mill sludge on nutrient leaching and soil phytotoxicity, and
2. appropriate application rates for minimizing nutrient loss, eliminating soil phytotoxicity and generating switchgrass growth.

Materials and Methods

Mine soil was collected from an abandoned mined land (AML) site in Upper Dauphin County, PA for use in the greenhouse. This material was characterized as coal refuse, the waste from processing and cleaning coal. Samples were air dried and passed through a 10-mm sieve prior to any amendment application. Soil characteristics were measured by the Agricultural Analytical Services Laboratory at Penn State University. Initial mine soil pH was 4.2 with extremely low bulk electrical conductivity (90 mS cm^{-1}). Nutrient levels were also extremely low with concentrations of 2 mg P kg^{-1}, 33 mg K kg^{-1}, 26 mg Mg kg^{-1}, and 108 mg Ca kg^{-1} and a moderately low CEC (7.3 cmolc kg^{-1}). Trace metals, such as Cu and Zn, were present in low concentrations. Organic C was 7.3%, but this was almost entirely coal C.

Amendment materials used in the greenhouse experiment included paper mill sludge (PMS), poultry layer manure and composted layer manure. Short fiber PMS was collected from the American Eagle recycled paper mill in Tyrone, PA. Layer manure was obtained from the high rise caged layer production facility operated by the Poultry Science Department at Penn State University, University Park, PA. Compost used in the greenhouse experiment was produced at the Organic Materials Processing and Education Center at Penn State University. Fresh poultry layer manure (from the same source as above), leaves collected in autumn, and ground wood were blended to obtain a C:N ratio of 20:1 then placed in a windrow and turned four times in the first 30 d. Water was added to the windrow one day before the first two turnings. After 30 d the windrow was reconfigured into a static pile for a 60 d curing period. Finished compost was screened 90 d later and stored for use in this experiment.

Table 2-1. Amendment application rates added to 5.76 kg mine soil and associated C, N, P and K added with each treatment.

Treatment	Additions to mine soil				
	Material	C	N	P	K
	g kg^{-1} dry wt.				
No amendment	—	—	—	—	—
Lime+fertilizer					
Limestone	5.00	0.61	—	—	—
NH_4NO_3	0.17	—	0.06	—	—
TSP	0.15	—	—	0.07	—
KCl	0.15	—	—	—	0.08
0.5x compost	15.0	5.1	0.36	0.10	0.17
1.0x compost	30.0	10.2	0.73	0.21	0.34
1.5x compost	45.0	15.3	1.09	0.31	0.51
7:1 manure	10.0	2.51	0.37	0.11	0.09
20:1 manure+PMS					
layer manure	10.0	2.51	0.37	0.11	0.09
paper mill sludge	15.0	5.00	—	—	—
30:1 manure+PMS					
layer manure	10.0	2.51	0.37	0.11	0.09
paper mill sludge	26.0	8.00	—	—	—
40:1 manure+PMS					
layer manure	10.0	2.51	0.37	0.11	0.09
paper mill sludge	38.0	12.00	—	—	—

Amendment materials were mixed with mine soil to give three rates of compost addition and 4 rates of manure+PMS addition (Table 2-1). The base compost addition (1x) was the amount of compost necessary to increase soil organic C by 1%. Compost was also added at 0.5 (0.5x) and 1.5 (1.5x) times the base rate. In the four manure+PMS treatments, a constant rate of manure was combined with increasing amounts of PMS to produce mixture C:N ratios of 7:1 (manure only), 20:1, 30:1 and 40:1. The manure addition in each treatment provided a total N addition equivalent to that which was added in the 0.5x compost rate. Additional treatments included a negative control (no amendment) and a positive control (traditional reclamation) of ground limestone and inorganic fertilizer. All treatments were mixed with 5.76 kg mine spoil material and poured into 30 cm tall polyvinyl chloride (PVC) columns measuring 15 cm in diameter. A flat PVC plate with a nipple in the center was glued to the bottom of each column and connected to PVC tubing that drained to a 1 L glass bottle for leachate collection. Inner surfaces of the columns were lined with Teflon to minimize any PVC constituent interference. Quantities of amendment materials added and resulting additions of C, N, and P are given in Table 1. Columns were placed in the greenhouse in a randomized complete block design with three replications for each treatment; ambient conditions included 16 h of light per day (0600-2200) and a constant temperature of $22 \pm 3\ ^{o}C$.

Each column was planted with 40 switchgrass seeds (*Panicum virgatum* L.) and watered with de-ionized water as needed to maintain moisture for plant growth only. Following a 90 d establishment period switchgrass was harvested once every 30 d for a total of six harvests. Clippings were dried for 48 hours at 60 ^{o}C, weighed, and ground (< 1 mm). A composite sample of all six cuttings was obtained by determining the proportion of each cutting contributing to the total yield and adding that amount to the composite 5 g oven-dry sample. Composite samples were analyzed for total N, P and K. Tissue concentrations of Ca, Mg, S, Al, Fe, Mn, B, Cd, Co, Cu, Mo, Na, Ni, Pb and Zn were measured with inductively coupled plasma (ICP) atomic emission spectroscopy after initial microwave digestion with HNO_3. Yield data were collected by measuring the mass of the oven dry matter harvested from each column.

One day prior to leaching, column moisture levels were brought to 80% of field capacity by weighing and adding necessary water. Columns were intentionally leached every 30 d for a total of six leaching events by adding de-ionized water in 50 ml increments to the mine soil surface every 30 minutes until 700 to 1000 ml of leachate was collected. Leachates were analyzed for pH and electrical conductance (EC). Al, Ba, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Pb, S, and Zn were analyzed by ICP. Organic C was measured by acidification and combustion on a Shimadzu Analyzer TOC 5000-A. Total N and total P were measured using in-line digestion followed by flow injection colorimetry and NO_3^- and NH_4^+ were analyzed using flow injection colorimetry on a Lachat QuickChem FIA+8000 Analyzer (Lachat Instruments, 2003a, 2003b, 2003c, 2007b). Nutrient quantities were calculated by multiplying the volume of leachate collected by the concentration measured. At the conclusion of the experiment, mine soil blends in each column were analyzed for pH (1:1 in water), total C and total N (combustion on a Fisions NA 1500 Elemental Analyzer), NH_4^+ (specific ion electrode) and available P, K, Ca and Mg (Mehlich-3 ICP).

Statistical analysis was performed using SAS 9.1 statistical software (SAS Institute, 2003). Analysis of Variance (ANOVA) and Fisher’s Protected Least Significant Difference (LSD) were used for mean comparisons. Single degree of freedom orthogonal contrasts compared groups of treatments. Repeated measures were used to determine the effects of time on leachate, soil and switchgrass yields. Covariance structures were determined for each repeated measures analysis by using the structure with the lowest Akaike's Information Criteria (AIC). Treatment effects were significant if $Pr > F > 0.05$ for switchgrass yield data and $Pr > F > 0.10$ for leachate, soils and tissue data to protect against a Type II error. In this experiment, a Type II error of concluding there was no difference when in fact there was one was considered to be the more serious error. For example, a conclusion that there was no difference in NO_3^- leaching among treatments could have more serious environmental implications than the alternative conclusion of finding treatment differences.

Results and Discussion

Leachate chemistry

Nitrogen

Very little N in any form was leached from unamended spoil (negative control) or from lime+fertilizer (positive control) amended spoil over six months of leaching (Table 2-2, Fig. 2-1). The net negative loss of N from unamended and lime+fertilizer columns suggests that native soil N was mineralized, which has been observed in other lab incubations (Table 2-1, 2-2) (Busscher et al., 1999; Douglas and Magdoff, 1991). Amendments with fresh poultry manure resulted in losses of 438, 192, and 110 mg column^{-1} of total N, NO_3^- and NH_4^+, respectively. This represents 21% of the total N originally added, with inorganic losses accounting for 15% of lost N. Cabrera et al. (1993), however, reported N losses of 60% from pelletized poultry manure treatments after 35 d of incubation, which, although much higher than losses measured in my experiment, demonstrate that manure has a high potential for N loss by leaching.

Even though the total amount of N added with the compost treatments was two to three times greater than N added with the lime+fertilizer treatment, the quantity of total N, NO_3^- and NH_4^+ leached from compost amended spoil did not differ from either of the control treatments. Increasing the amount of compost added did not increase N leaching, in fact relative to the quantity of N added total N leaching was decreased. The 0.5x and 1.0x compost treatments lost 6% and 3%, respectively, of original N added, while only 2.3% of added N was lost by leaching from the 1.5x compost treatment. Similarly, all compost treatments lost < 1% of added N as inorganic NO_3^- or NH_4^+, demonstrating that large quantities of mature compost can be applied to soil with almost no risk of N leaching from the soil.

Flavel and Murphy (2006) observed a 12-fold decrease in total N losses from soil incubated with pelletized poultry manure (60 mg N kg^{-1}) compared to compost (5 mg N kg^{-1}) after 150 d, a difference very similar to that observed in my experiment. The losses I measured are even lower than those found by Adegbidi et al. (2003) in their greenhouse study testing large applications of composted poultry manure for willow production as a bioenergy crop. With a compost application rate of 35 g kg^{-1}, they measured losses of 32% of originally added N after 36 weeks, which was still considerably less than the 57% N loss from non-composted sewage sludge. Therefore they concluded that while these rates were twice as large as necessary for high willow production, compost was still more efficient at retaining N than non-composted materials. Other studies have measured inorganic N losses of 11 to 29% of original total N from incubated compost treatments and, although higher than N losses measured in my experiment, were still much smaller than losses from non-composted poultry manure (Hadas and Portnoy, 1994; Castellanos and Pratt, 1981). Conversely, Douglas and Magdoff (1991) observed N immobilization from composted manure and sewage sludge treatments in an incubation study, but this did not seem to be a problem in this experiment.

Increasing the C:N ratio of fresh manure with PMS addition also reduced N leaching, but not as effectively as composting. Adjusting the C:N ratio from 7:1 to 30:1 reduced total N, NH_4^+ and NO_3^- leaching by 0.62x, 0.20x, and 0.12x, respectively, a more substantial decrease than was achieved with the 20:1 or 40:1 C:N ratio (Table 2-2).

In a greenhouse column experiment testing NH_4NO_3 or poultry manure mixed with PMS in a 20:1 C:N ratio, Busscher et al. (1999) also found that the addition of poultry manure to PMS worked better to reduce N leaching than adding inorganic fertilizer to the manure. A C:N ratio of 40:1 resulted in a small increase in leaching of total N, NO_3^- and NH_4^+ compared to the 30:1 C:N ratio. The 40:1 C:N treatment, which received 1.5x more C than the 30:1 treatment, would have been expected to immobilize more N given the high C:N ratio (Vigil and Kissel, 1991; Parker and Sommers, 1983). It is unclear why this trend was observed, but could be related to a physical or chemical inhibition of microbial activity caused by the paper mill sludge at high application rates. All three adjustments of poultry manure C:N, however, reduced N leaching compared to fresh manure, confirming the hypothesis that C:N adjustment is an effective way to control N losses by leaching under intense greenhouse conditions.

Additionally, the 30:1 C:N ratio did not leach more total N than any of the compost treatments, although this was not the case for leached NO_3^- or NH_4^+ due to the large proportion of organic, rather than inorganic, N leached from compost. While compost treatments leached very minimal quantities of inorganic N, it is important to consider that as much as 40% of manure N can be lost in the composting process, mostly as NH_3 (Eghball et al., 1997). Even with these losses, compost has such minimal N losses in my experiment due to N transformations during the composting process, which stabilized N into complex, humic-like molecules (Amlinger et al., 2003). Given that poultry manure mixed with PMS in a 30:1 C:N ratio does not lose more total N than compost treatments, however, the manure+PMS treatment appears to be composting *in situ*, or transforming N into more recalcitrant forms, indicating that composting manure prior to amendment application in soil may not be necessary to achieve the same N transformations as occur in a compost pile.

Losses of all forms of N were extremely low throughout the six leaching events for the unamended and lime+fertilizer amended soils (Fig. 2-1). Concentrations of NO_3^- leached from these treatments never exceeded the EPA drinking water standard of 10 mg L^{-1} throughout the experiment (USEPA, 2008). In compost amended columns, initial N loss was also very low. Losses were predominantly organic N and the quantity of total N decreased with time, reflecting the more substantial organic N content of the compost material.

In the manure+PMS 7:1 treatment, leaching of all forms of N subsided after the first three leaching events and NO_3^- concentrations remained below 10 mg L^{-1} thereafter (Fig. 2-1). In the first leaching event, 185 mg total N $column^{-1}$, or 7% of added N, was leached from mine soil amended with fresh manure, almost entirely as inorganic NH_4^+ and NO_3^-. This initial rapid loss of NH_4 was also reported by Cabrera et al. (1993) after 3 d of incubation of pelletized poultry manure. Hanselman et al. (2004) recorded even higher mineralization and leaching of poultry manure N (38 – 47% of added organic N) within the first 3 d of *in situ* and lab incubations. Chae and Tabatabai (1986), however, reported very little N mineralization of poultry manure during the initial stages of incubation (up to four weeks), but saw a marked increase in mineralization rates thereafter. The higher initial rates of release from fresh manure in my experiment could be attributed to the sandy and rocky texture of the soil, as opposed to the loamy and silty textures used in the aforementioned study. Chae and Tabatabai (1986), however, did conclude that poultry manure had greater mineralizable N than other manures tested in the study, including cow, hog and horse.

The quantity of NO_3^- leached was initially low in manure+PMS treatments then increased before dropping again after the third leaching event (Fig. 2-1). This trend suggests some initial N immobilization followed by a shift toward more mineralization and nitrification. After the third leaching event, concentrations in all manure treatments, regardless of C:N ratio, were less than 10 mg NO_3-N L^{-1}. Busscher et al. (1999) also observed a decrease of NH_4^+ after 22 d and NO_3^- after 12 d in a greenhouse study of poultry manure mixed with PMS in a 20:1 ratio. Their study, however, used weekly leaching events and therefore had a more intense schedule than the monthly leaching events in this experiment.

Table 2-2. Cumulative quantities of major nutrients collected in leachate from six leaching events. Values are the means of three mine soil columns.

		Cumulative leachate analysis			
Treatment	C	N	NH_4^+	NO_3^-	P
	mg column^{-1}				
No amendment	493	109	0.03	0.13	0.30
Lime+fertilizer	422	115	0.01	7.08	0.30
0.5x compost	520	122	0.17	0.66	0.45
1.0x compost	652	127	2.85	0.72	1.48
1.5x compost	465	142	0.14	1.67	3.38
7:1 manure	465	438	110	192	12.0
20:1 manure+PMS	455	271	53.1	114	0.86
30:1 manure+PMS	497	167	21.8	23.4	0.61
40:1 manure+PMS	1000	235	78.7	48.1	1.99
Analysis of variance					
			p Values		
Source of variation					
Treatment	NS[†]	*	*	*	*
Single degree of freedom contrasts					
pos-cntrl v. compost	—[‡]	NS	NS	NS	NS
pos-cntrl v. man+PMS	—	*	*	*	*
comp rate v. man+PMS	—	*	*	*	*
compost rate	—	NS	NS	NS	*
man+PMS C:N linear	—	*	NS	*	*
man+PMS C:N quadratic	—	*	*	*	*
Mean comparisons					
Protected $\text{LSD}_{0.10}$	—	60.6	46.2	48.2	2.23

* Significant at α = 0.10

[†] Not significant at α = 0.10

[‡] Not analyzed due to no significant treatment differences

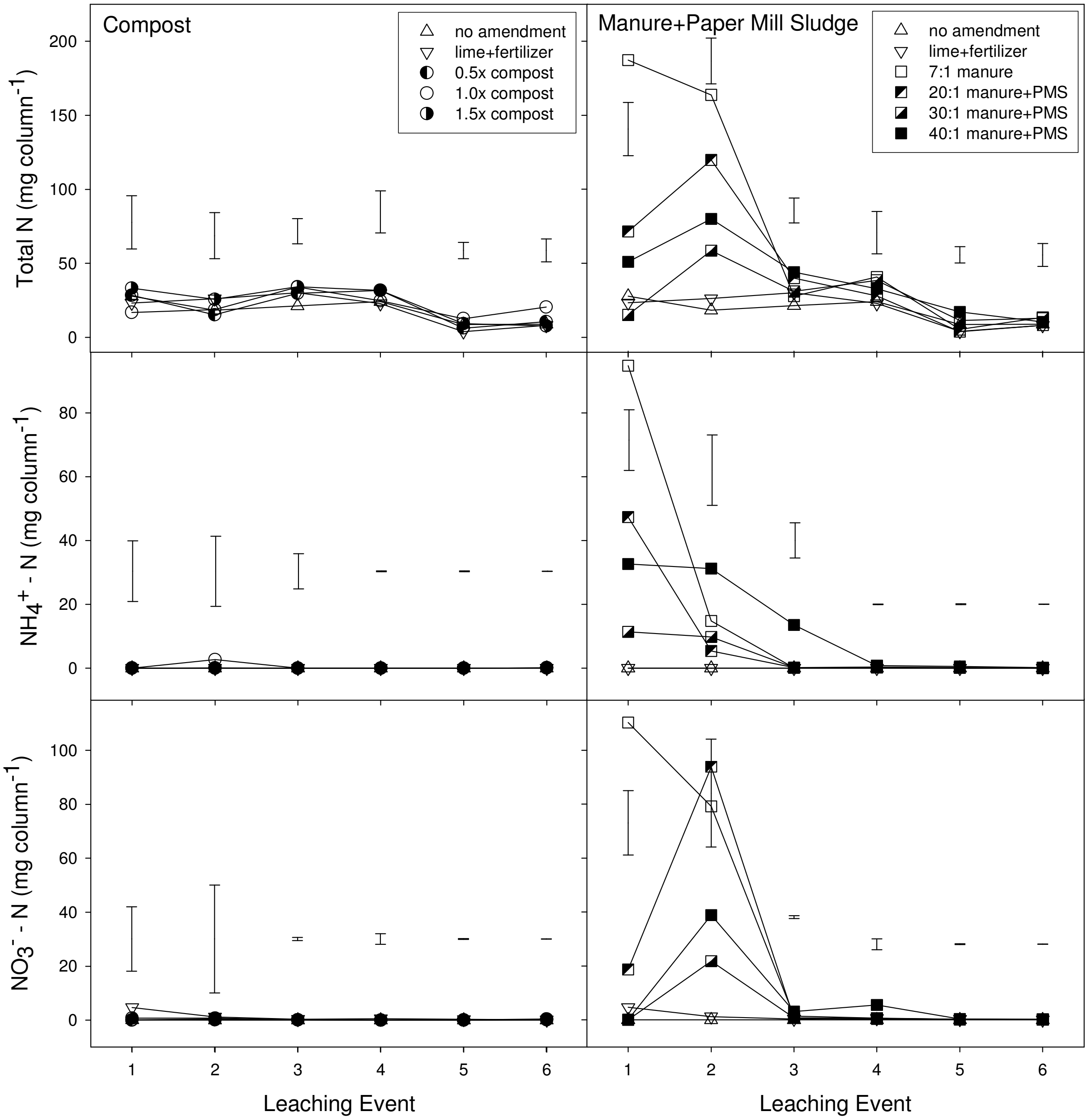

Figure 2-1. Quantity of total N, NH_4-N and NO_3-N leached from mine soil amended with compost or manure. Error bars indicate width of $LSD_{0.10}$ for each leaching event.

Phosphorus

Both the unamended and lime+fertilizer amended columns leached minimal total P throughout the experiment, demonstrating no risk of P leaching with conventional fertilization of these soils, even under intense leaching conditions (Table 2-2). Cumulatively, the 1.5x compost leached 3.38 mg P column^{-1}, 7.5 and 2.3 times more P than was lost with the 0.5x and 1.0x compost treatments, respectively, demonstrating a higher rate of P loss with large applications of compost. Eghball and Gilley (1999) reported higher runoff losses of P from composted compared to fresh beef manure, suggesting that P loss from large compost applications could be an environmental concern. At each leaching event, however, mine soil amended with compost leached less than 1 mg total P column^{-1}, or less than half a percent of originally added P; these losses of P were consistent over time (Fig. 2-1, Table 2-1).

Eleven times more total P was leached from the fresh manure treatment than from all other manure+PMS treatments; this represents a loss of 12 mg P column^{-1}, but < 2 % of originally added P (Table 2-1, 2-2). Other studies, such as a column experiment by McDowell et al. (2001), have measured greater total P leachate losses. In their study, total P concentrations in leachate were 3.6x larger 3 weeks after dairy manure application compared to P concentrations prior to application. These losses, however, were dependent on the concentration of soil P, or the degree to which soil was saturated with P, prior to manure amendment. Given that the mine spoil had very low initial P levels, it is not surprising that minimal P would be lost by leaching, even with a fresh manure treatment.

All treatments with added paper mill sludge greatly reduced the amount of P leached from the mine soil. Manure+PMS treatments leached less than 2 mg column^{-1} over the course of the experiment, similar to quantities lost from compost treatments (Table 2-2). The 40:1 C:N blend leached more P in the initial leaching event; in the second event, the 40:1 and 20:1 blends leached more P than the 30:1 blend. Quantities leached did not differ among manure+PMS treatments throughout the rest of the experiment. It seems likely, given the high C:P ratios of these treatments (68:1 to 132:1), that there was some immobilization of P that contributed to minimizing P losses from these treatments (Enwezor, 1976). Furthermore, the addition of $CaCO_3$ in the PMS, combined with the rapid increase in soil pH following amendment addition (5.1 to 7.0), likely resulted in the precipitation of Ca phosphates, which further reduced the amount of soluble P available for leaching (Havlin et al., 1999).

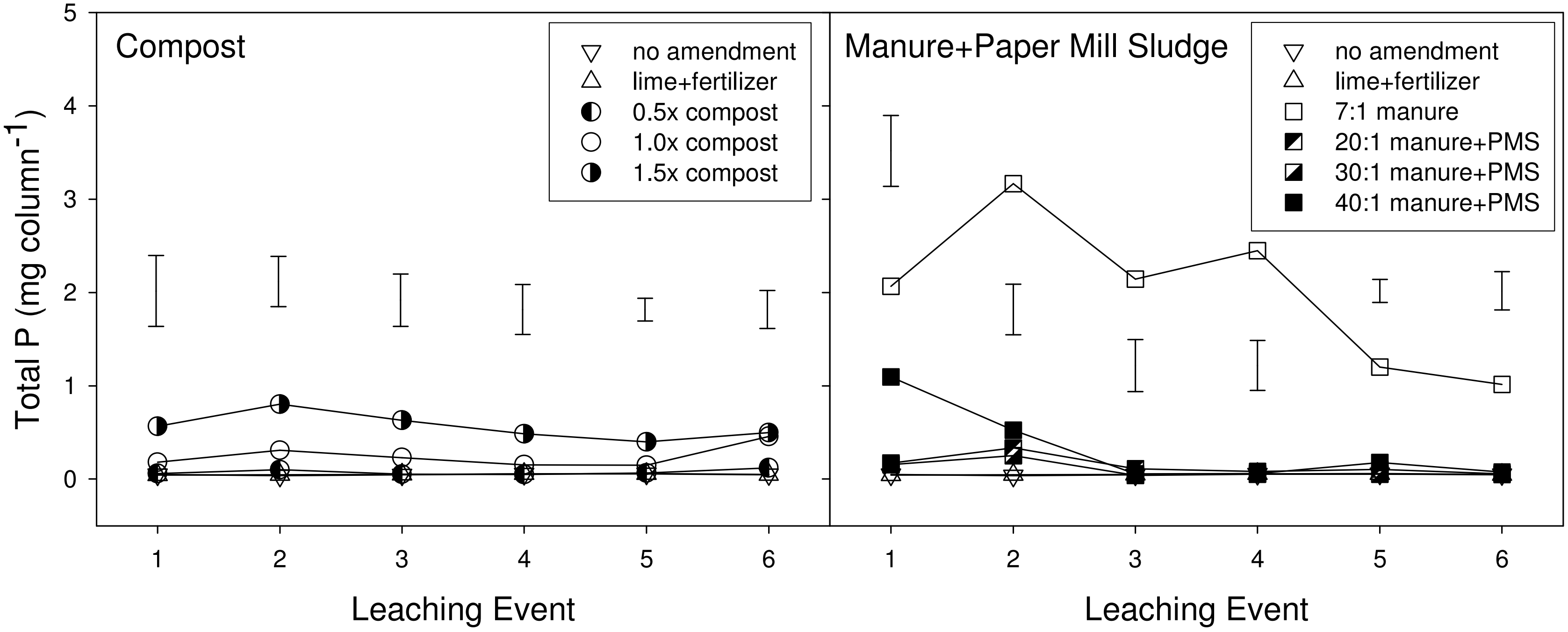

Figure 2-2. Quantity of total P leached from mine soil columns amended with compost or manure. Error bars indicate width of $LSD_{0.10}$ for each leaching event.

Other nutrients

As would be expected, more Ca was lost from the lime and fertilizer control than from any other treatment due to the large quantity of Ca added as lime (5 g kg^{-1}) (Table 2-3). Similarly, a large quantity of Mehlich-3 extractable Ca (2120 mg kg^{-1}) was measured in the soil at the conclusion of the experiment, showing that while Ca was lost by leaching, much of the mobilized Ca remained in the soil material (Table 4). Compost treatments did not differ in Ca leaching based on application rate, a result similar to that reported by von Willert and Stehouwer (2003) in a column experiment in which coal mine spoil was amended with various combinations of biosolids compost, lime, and gypsum. In their study, however, they found little difference in Ca leaching between lime and compost amended spoil. In my experiment, lime+fertilizer treatments leached significantly more Ca than compost treatments. Extractable soil Ca data showed more Ca in the 1.5x and 1.0x compost treatments, reflecting the larger quantities of Ca added with increasing rates of compost. Although the manure+PMS 40:1 treatment leached less Ca than lime+fertilizer, it lost more Ca than all other organic treatments, likely due to the large quantities of $CaCO_3$ in the paper mill sludge. Extractable soil Ca data revealed no difference in Ca accumulation in the soil, suggesting that more Ca was applied with the 40:1 treatment than could be sorbed by the soil, resulting in leaching losses.

Among compost treatments, quantities of K leached were proportional to the amount added with each treatment (Table 2-1, 2-3). Conversely, the 7:1 manure amendment leached more K than the 20:1 and 30:1 manure+PMS treatments even though the same quantity of K was added with manure in each treatment. Although some treatment differences for other nutrients, such as S and Na, were observed, these do not appear to be of great consequence, as quantities leached were not abnormally high so as to cause environmental or crop concerns.

Metals

Although there were differences in leaching by treatment of some metals, quantities were extremely low and at no point in the experiment did concentrations exceed EPA drinking water standards (USEPA, 2008). There were no differences in the leached quantities of Ba, Cd, Cr, Mo, Ni, and Pb among any treatments (Table 2-3). These results are similar to those found by Sommers et al. (1979), where the leaching of metals from incubated soils amended with sewage sludge was very minimal. In addition, PMS and poultry manure, which contain comparable concentrations of heavy metals, tend to have lower concentrations than sewage sludge (Bellamy et al., 1995). Furthermore, no studies reviewed reported problems with heavy metal leaching in association with any materials used in this experiment when applied to soil on a one-time basis. Therefore, there is no concern of heavy metal contamination to the environment from applying any of these amendments to mine spoil.

Table 2-3. Cumulative quantities of metals and nutrients collected in leachate from six leaching events. Values are the means of three minespoil columns. Ba, Cd, Cr, Mo, Ni, and Pb were not significant at $\alpha = 0.10$ and therefore excluded from the table.

	Cumulative leachate analysis									
Treatment	Al	Ca	Cu	Fe	K	Mg	Mn	Na	S	Zn
	mg column^{-1}									
No amendment	1.24	152	0.01	0.02	5.5	10.7	0.16	4.40	151	0.16
Lime+fertilizer	1.22	663	0.01	0.02	15.7	12.3	0.00	3.20	358	0.02
0.5x compost	9.92	237	0.05	9.07	34.7	32.2	0.02	33.9	235	0.02
1.0x compost	3.51	342	0.06	5.61	86.6	50.2	0.01	64.3	276	0.02
1.5x compost	3.56	391	0.09	5.53	137	59.0	0.00	91.5	304	0.02
7:1 manure	1.50	322	0.04	0.54	136	43.7	0.22	94.6	208	0.04
20:1 manure+PMS	1.12	408	0.05	0.03	76.4	28.8	0.04	55.5	124	0.02
30:1 manure+PMS	1.02	333	0.03	1.42	40.7	18.8	0.02	39.0	102	0.02
40:1 manure+PMS	1.28	517	0.08	4.25	111	47.0	0.07	66.1	102	0.04
	Analysis of variance									
	p Values									
Source of variation										
Treatment	*	*	*	*	*	*	*	*	*	*
	Mean comparisons									
Protected LSD$_{0.10}$	1.41	96.1	0.03	3.61	47.3	22.4	0.06	23.0	54.0	0.04

* Significant at $\alpha = 0.10$

Soil chemistry

Soil pH

Initial mine soil conditions were inhibiting to plant growth, as evidenced by lack of vegetation at the collection site as well as the initial soil analysis, most notably the very low pH of 4.2. The unamended mine soil pH remained low throughout the experiment, increasing only to 5.2 after six intensive leaching (Table 2-4). Manure alone raised soil pH to 6.0 at the end of six months, likely a result of the hydrolysis of urea and $CaCO_3$ in the manure (eggshells and hen feed) and the low buffering capacity of the soil (Cabrera et al., 1993). Lime and fertilizer amendment caused the largest increase in soil pH (7.9), reflecting the large initial lime addition of 5 g kg^{-1}.

However, all organic treatments also substantially increased soil pH and effectively ameliorated phytotoxicity as evidenced by switchgrass growth. Calcium carbonate equivalency (CCE), or the quantity of alkalinity added expressed as Mg CaCO3 ha^{-1}, for the organic treatments were high, with compost contributing approximately 20 to 40 Mg ha-1 and manure+PMS treatments contributing 50 to 80 Mg ha^{-1}. Among compost treatments, final soil pH differed for each rate of application: 6.5, 7.0, and 7.2 for the 0.5x, 1.0x and 1.5x rates, respectively. In a field experiment on a loamy soil, Aggelides and Londra (2000) measured significant increases in soil pH (6.8 to 7.2) and CEC (14.4 to 22.6 cmolc kg^{-1}) proportional to the rate of compost addition. While a similar final pH was observed in compost treated columns in this study, the CEC was effectively increased only with the 1.5x compost treatment. Furthermore, Eghball and Gilley (1999) measured a soil pH increase of 5.2 to 7.2 with the addition of fresh or composted beef manure in a field setting, suggesting that both materials are capable of raising soil pH.

The three manure+PMS treatments increased soil pH from 7.5 to 7.7 with increasing C:N ratio (Table 2-4). Not only was mine spoil pH increased further by the co-application of manure and PMS, but higher mineralization rates measured from these treatments likely had acidifying effects on the soil, implying that the liming effect of these treatments was actually greater than that of lime alone (Stamatiadis et al. 1999; Beegle and Lingenfelter, 1995). Regardless of composting or fresh application of manure with PMS, these treatments effectively raised soil pH to levels more suitable to plant growth.

Table 2-4. Analysis of amended soil in columns at the conclusion of the experiment**.** Values are the means of three minespoil columns.

	Final soil analysis												
Treatment	pH	N	C	NH_4^+	P	K	Mg	Ca	K	Mg	Ca	acidity	CEC
		g kg^{-1}		mg kg^{-1}					cmolc kg^{-1}				
No amendment	5.2	4.30	73.3	1.33	3.71	21.7	39.0	501	0.1	0.3	2.5	6.7	9.6
Lime+fertilizer	7.9	4.37	78.2	0.83	17.4	32.2	35.7	2120	0.1	0.3	10.6	0.0	11.0
0.5x compost	6.5	4.80	80.1	1.09	37.7	40.5	92.9	1250	0.1	0.8	6.3	2.1	9.3
1.0x compost	7.0	5.10	88.2	1.44	75.0	49.8	111	1490	0.1	0.9	7.5	0.0	8.6
1.5x compost	7.2	5.50	89.9	1.59	131	62.7	145	1960	0.2	1.2	9.9	0.0	11.2
7:1 manure	6.0	4.70	74.3	1.61	61.4	30.9	54.0	820	0.1	0.5	4.1	2.8	7.4
20:1 manure+PMS	7.5	4.63	79.0	1.43	77.3	36.7	63.3	1760	0.1	0.5	8.8	0.0	9.5
30:1 manure+PMS	7.6	4.67	87.4	1.48	56.1	30.3	60.6	1920	0.1	0.5	9.7	0.0	10.3
40:1 manure+PMS	7.7	4.63	86.9	1.37	60.7	26.1	61.3	2320	0.1	0.5	11.7	0.0	12.3
	Analysis of variance results												
	p Values												
Source of variation													
Treatment	*	*	*	*	*	*	*	*	*	*	*	*	*
	Mean comparisons												
Protected $LSD_{0.10}$	0.13	0.15	6.83	0.23	29.9	5.32	14.2	490.4	0.027	0.081	1.78	0.31	1.85

* Significant at $\alpha = 0.10$

Soil N

Total N in the final mine soil was greatest in the compost treatments, approximately one third of added N was found in the soil at the end of the experiment (amendment plus initial soil N) (Table 2-4). These results, combined with minimal N leaching and high rates of application, confirm that compost treatments are extremely efficient at storing N in the soil despite intense leaching conditions. Some studies have suggested that, although compost contributes to soil N, the high C:N ratio of the material causes the N to be unavailable to plants (Cooperband et al., 2002). This does not appear to be the case here, though, given the large switchgrass growth recorded with these treatments and growth response to increasing compost application rate. There was no difference in soil total N among fresh manure treatments, regardless of C:N ratio, but these treatments all retained more N than the lime+fertilizer treatment. Eghball (2000) also measured similar N values in a field soil 1 yr after amending soil with beef manure.

Soil C

The 1.0x and 1.5x compost treatments, along with the manure+PMS 30:1 and 40:1 treatments had the highest concentrations of total C remaining in the soil at the end of the experiment (average 88 g C kg^{-1}) (Table 2-4). These results reflect the large quantities of C added with these treatments, suggesting C has been effectively retained in the soil with these treatments. It is important to note that roots in the columns were root-bound and all roots could not be removed prior to soil C analysis, making these soil C results inconclusive with respect to quantities of C sequestered. Fresh manure, manure+PMS 20:1 and the two control treatments retained the least amount of C, suggesting that insufficient C (0 – 5 g kg^{-1}) was added with these treatments to impact organic matter content of the mine soil. Alternatively, a 360 d incubation study by Pascual et al. (1999) demonstrated long-term soil benefits of adding fresh sewage sludge to soils as opposed to composted sludge, wherein properties such as soil organic matter and microbial biomass were measurably greater in soils amended with fresh material even after 8 yr. This would support the use of manure+PMS in 30:1 or 40:1 C:N ratios over composted manure in regard to improving both soil C and long-term soil quality.

Soil P

Soils treated with the 1.5x compost amendment had Mehlich-3 extractable P concentrations twice as large as all other organic amendments and almost 8x more soil P than lime+fertilizer (Table 2-4). P concentrations were significantly elevated in all treatments with manure, regardless of application method, compared to unamended or lime+fertilizer treatments. A linear relationship was found between P added with treatments and soil P at the end of the experiment ($r^2 = 0.66$; $p = 0.0080$). Cooperband et al. (2002) also noted this linear relationship in a field study on a silt loam soil, demonstrating that the quantity of P added was more important in determining soil P than the source (fresh or composted). They did, however, measure three times more soil P in soils amended with composted verses raw poultry manure. These results also concur with those of Eghball and Power (1999) who found a strong correlation between soil test P and total P in manure or compost treatments in a field study using composted or raw cattle manure. Furthermore, they measured soil P at 116 mg kg^{-1} for fresh manure treatments and 127 mg kg^{-1} for composted manure after one year, values similar or greater than those measured with the intensive leaching schedule of my greenhouse experiment. Based on these results, the buildup of soil P from a one-time application of these amendments, coupled with the minimal leaching losses of P, confirm that these treatments would not pose a threat of P release into the environment (Sharpley et al., 1996).

Other nutrients

At the end of the experiment, compost treatments had greater concentrations of K and Mg remaining in the soil than other treatments, likely providing sustained nutrients for plants (Table 2-4) (Sims, 1990). These results differ from those of Hartl et al. (2003), who found no difference in soil nutrient concentrations between soils receiving composted biowaste or no organic inputs in a field study; however, in a three year field study using these amendments, soil nutrient concentrations were significantly greater in mine soil amended with organic amendments compared to lime+fertilizer (see chapter IV). Manure use has also been identified to increase soil Mg and K concentrations relative to inorganic fertilizer; however, this was not the case in this experiment. The lack of difference in soil nutrients with manure treatments shows there is no risk of excessively high soil concentrations with the use of manure that could potentially be leached to groundwater (Edmeades, 2003).

Switchgrass

Yield

Unamended mine soil columns produced almost no switchgrass growth (Table 2-5, Fig. 2-3). The lime+fertilizer control, however, did improve switchgrass growth beyond that of the unamended soil. The 0.5x compost treatment also did not significantly improve switchgrass growth over lime and fertilizer; the 1.0x and 1.5x compost treatments, however, significantly increased switchgrass growth and there was a significantly linear response to compost rate. Even under highly acidic soil conditions ($pH < 3$), tall fescue yields of 28.1 g were attainable with a high rate of biosolids compost addition (68.8 g kg^{-1}), demonstrating that composts can effectively improve biomass yields, especially when applied in large quantities to impaired soils (von Willert and Stehouwer, 2003).

Fresh manure alone produced almost 2.5 times more switchgrass than the 1.5x compost treatment, showing an even greater capacity of this treatment to support vegetation under an intense harvest schedule (1 harvest per mo). Field studies have recorded slightly lower yields with manure than were measured here, but yields are nonetheless improved with manure. A study involving sewage sludge applied to a corn crop measured a 1.3-fold increase in corn yield after 12 wk compared to plots amended with NH_4NO_3, demonstrating the ability of fresh organic wastes to improve yields (Sims and Boswell, 1980).

Increasing the C:N ratios of manure+PMS improved switchgrass growth even further, with the 40:1 C:N producing almost 1.3 times more switchgrass than the 30:1

manure plus paper mill sludge treatment. Biomass yields from the 40:1 treatment were also 3.6x larger than the 1.5x compost treatment, 14x larger than lime+fertilizer amended columns, and 47x larger than unamended soils. The linear yield increase with increasing compost addition and with increasing PMS addition demonstrate these organic amendments have the potential to vastly improve soil quality of mine soils and to transform phytotoxic mine soils into highly productive systems.

Tissue analysis

With the exception of the unamended mine soil, nutrient and trace metal concentrations were within a normal range for plant growth, confirming that these treatments effectively remediated phytotoxic conditions at the site (Table 2-6). Higher N concentrations were observed in treatments with fresh manure, perhaps a result of higher soil NH_4^+ and NO_3^- (Sims and Boswell, 1980). Conversely, plants grown in compost treated mine soil had higher tissue P concentrations than those grown in fresh manure, which corresponds to larger initial P additions and soil test P levels (Table 2-1, 2-4).

Table 2-5. Biomass production at each cutting and cumulative biomass (Cum) collected across all six cuttings by treatment. Values are the means of three mine soil columns.

	Cutting						
Treatment	1	2	3	4	5	6	Cum
	mg column^{-1}						
No amendment	0.20	0.03	0.02	0.09	0.22	0.36	0.91
Lime+fertilizer	1.24	0.54	0.36	0.20	0.22	0.45	3.02
0.5x compost	0.51	0.39	0.70	0.66	0.81	0.79	3.86
1.0x compost	1.93	1.44	1.44	0.64	0.54	1.99	7.99
1.5x compost	3.78	1.72	1.59	1.09	1.21	2.34	11.7
7:1 manure	5.68	4.02	6.41	4.81	3.10	4.47	28.5
20:1 manure+PMS	8.18	3.47	6.28	4.64	2.57	1.07	29.2
30:1 manure+PMS	7.96	5.92	6.94	4.93	2.58	4.68	33.0
40:1 manure+PMS	9.45	7.99	8.20	6.94	3.69	6.64	42.1

Analysis of variance results

				p Values			
Source of variation							
Treatment	**	**	**	**	**	**	**
Single degree of freedom contrasts							
pos-cntrl v. compost	NS†	NS	NS	NS	NS	**	**
pos-cntrl v. man+PMS	**	**	**	**	**	**	**
comp rate v. man+PMS	**	**	**	**	**	**	**
compost rate	NS	NS	NS	NS	NS	**	**
man+PMS C:N linear	NS	**	**	**	NS	**	**
man+PMS C:N quadratic	NS	NS	NS	NS	NS	**	**

Mean comparisons

Protected $\text{LSD}_{0.05}$	3.56	2.17	1.31	1.55	1.25	1.17	4.37

** Significant at $\alpha = 0.05$

† Not significant at $\alpha = 0.05$

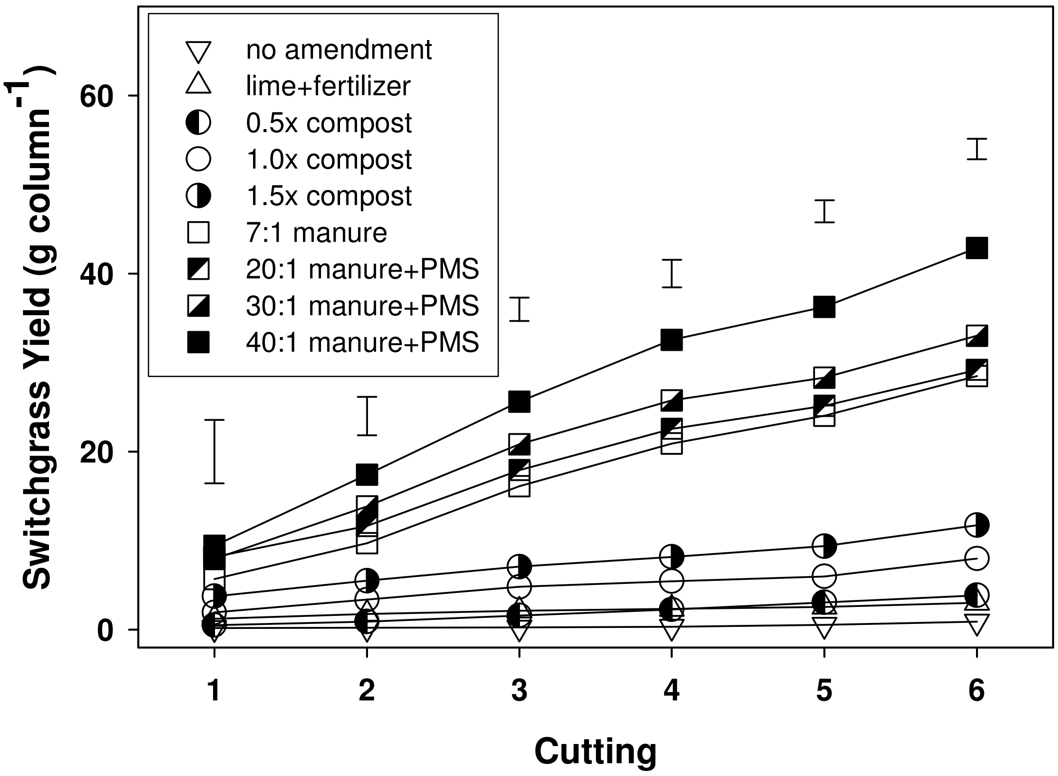

Figure 2-3. Cumulative switchgrass yield collected at each leaching event. Error bars indicate width of $LSD_{0.05}$ for each cutting.

Table 2-6. Switchgrass tissue analysis based on a composite sample collected from six cuttings. Values are the means of three mine soil columns. Al, Cd, Na and Ni were not significant and not included in this table.

	Cumulative tissue analysis												
	Macronutrients						Micronutrients						
Treatment	N	P	K	Ca	Mg	S	Fe	Mn	B	Co	Mo	Pb	Zn
	$g\ kg^{-1}$						$mg\ kg^{-1}$						
No amendment	13.9	1.15	14.8	3.43	1.32	1.80	66.2	87.4	6.96	—†	—†	—†	32.3
Lime+fertilizer	14.9	2.84	16.7	5.80	2.16	1.43	47.8	8.27	5.23	0.04	0.27	0.25	13.8
0.5x compost	12.2	3.82	15.3	4.14	2.42	1.51	65.6	49.6	12.3	0.05	0.25	0.25	25.3
1.0x compost	11.6	4.20	14.8	4.19	2.67	1.40	46.3	34.2	20.4	0.06	0.65	0.25	23.1
1.5x compost	12.5	4.29	18.2	4.14	2.65	1.35	49.6	26.8	18.8	0.04	0.96	0.25	27.5
7:1 manure	17.4	2.64	19.0	4.54	2.38	1.69	52.6	114	13.8	0.10	0.14	0.37	30.4
15:1 manure+PMS	16.1	2.94	18.1	5.30	2.70	1.62	44.3	31.6	9.33	0.04	0.37	0.25	22.7
25:1 manure+PMS	17.5	3.00	18.1	6.36	2.86	1.74	46.8	28.7	10.2	0.05	0.47	0.25	23.2
35:1 manure+PMS	16.7	2.83	17.3	6.48	2.86	1.66	45.3	27.9	9.74	0.04	0.68	0.25	22.8
Optimal levels‡	13.2	2.20	16.3	3.9	3.00	1.70	58.0	123	—§	—§	—§	—§	22
	Analysis of variance												
	p Values												
Source of variation													
Treatment	*	*	*	*	*	*	*	*	*	*	*	*	*
	Mean comparisons												
Protected LSD$_{0.10}$	2.06	0.361	2.22	0.780	0.321	0.213	11.1	12.5	2.51	0.033	0.174	0.057	4.93

* Significant at $\alpha = 0.10$

† Insufficient sample to run analysis due to poor switchgrass growth

‡ Data from Reid et al. (1992)

§ No data available

Conclusions

Results of this experiment clearly demonstrate that poultry manure can be applied to acidic mine soil to alleviate soil phytotoxicity and improve switchgrass growth. Traditional lime and fertilizer additions also ameliorated soil phytotoxicity and incurred minimal losses of nutrients by leaching, even under intense leaching conditions, but the treatment failed to promote vegetative growth in quantities necessary for intensive biofuel production. Leaching losses from fresh manure application (10 g kg^{-1}) were substantial (438 mg N $column^{-1}$). The environmental consequences of this N leaching outweigh the 9.5x yield increase over that measured with NH_4NO_3 fertilizer and lime, demonstrating that poultry manure alone is not adequate to meets the concurrent goals of minimal N leaching, improved soil quality, and high switchgrass yields. Alternatively, composting poultry manure prior to application substantially reduced N and P leaching losses while further improving switchgrass growth over simple lime+fertilizer additions, even under an intense leaching schedule. Composted poultry manure can therefore add nutrients to mine spoil and achieve large yields with essentially no risk of N leaching to the environment.

Adding manure+PMS in a 30:1 C:N ratio, however, not only minimized N and P leaching losses to the same extent as composted manure, but also promoted switchgrass yields that were over 4x larger than with the highest rate of compost tested (45 g kg^{-1}). Not only does this suggest that manure+PMS treatments would be better to obtain the high yields necessary for biofuel crop production, but the increased soil C and N levels indicate that the addition of these materials could promote a more sustainable system, another necessary component of intensive grass production on these sites. Given these promising results under more extreme conditions than would occur in the field, this approach to using poultry manure should be further investigated in a field experiment.

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Chapter III

Stable and labile nitrogen and carbon pools in coal mine soils amended with poultry manure

Introduction

Soil labile nitrogen (N) pools are susceptible to mineralization and subsequent leaching losses that can negatively impact water quality. In order for soils to effectively retain N, microbial cycling or abiotic reactions must transform N into more stable N pools (Kaye et al., 2002a; Kelly and Stevenson, 1987; Paré et al., 1998). Moreover, microbial activity plays a substantial role in preventing inorganic N loss and is therefore a crucial component of both retaining N and restoring nutrient cycling in nutrient depleted mine soils (Stark and Hart, 1997; Davidson et al., 1992). Abandoned mine soils, having extremely low nutrient concentrations, may also have a high capacity to retain N as long as adequate C is present to sustain microbial activity (Kaye et al., 2003; Ingram et al., 2005; Akala and Lal, 2001).

The use of compost to improve soil quality and minimize N loss by leaching has been well documented (Amlinger et al.,2003; Bernal et al., 1998; Gallardo-Lara and Nogales, 1987). An incubation study of compost found that the improvement of soil properties, such as hydraulic conductivity, water retention capacity, bulk density, porosity, and aggregate stability, were proportional to the rate of compost addition (Aggelides and Londra, 2000). Adding fresh manure to soil has also been shown to improve long-term soil microbial activity, and it has also been suggested that less processed materials, such as sewage sludges, provide a better microbial food supply

(Pascual et al., 1997; Gigliotti et al., 2002). But, excessive amounts of inorganic N are at risk for loss by leaching with the application of fresh manure, meaning that manure alone has minimal capacity to retain N in a stable form (Carpenter et al., 1998). Mixing manure with a high carbon (C) source, however, has shown potential for minimizing N leaching loss and improving soil properties (see Chapter II). While it appears the co-application of these materials significantly reduces the labile N pool, it is unclear how the stable N and C pools are affected and to what degree microbial activity is sustained with fresh, rather than composted, poultry manure application.

Incubation studies are ideal for examining N and C transformations and the formation of labile and stable N and C pools, as these measurements are taken without the influence of plants or environmental elements (Hart et al., 1994). Using this approach I designed two parallel laboratory incubation experiments to study how labile and stable N and C pools differentiate immediately following amendment application and one year after the same quantities of amendments were applied in the field.

In the year one study, fresh material was applied to mine soil in the lab and then incubated for one year. The goal of the year one study was to maximize mineralization and thereby measure the inorganic N leaching potential of mine soils immediately following application of lime and fertilizer, composted poultry manure, or fresh manure mixed with paper mill sludge. In addition, determining the stable N and C pools would assess the ability of these treatments to retain nutrients in the long-term, while the measurement of microbial biomass would give an indication of the microbial activity associated with these treatments.

In the year two study, samples were collected from field plots one year after amendments were applied in the same ratios. The goal of this study was to measure labile and stable N and C pools, comparing these values to those predicted by the year one experiment and assessing how they have changed after one year in the field. Also, a determination of the microbial biomass pool would help predict the long-term restoration of nutrient cycling in soils amended with these materials.

Materials and Methods

Year one soil samples

Soil material for the manufacture of the blends in the year one incubation experiment was collected from an abandoned mineland (AML) site in Schuylkill County, PA. Initial properties of the soil included a pH of 5.1, a very channery sandy loam texture, and a bulk density of 1.77 g cm^{-3}. Annual precipitation at this site was 132 cm. At each of three locations at the AML site, 75 cores (each core was approximately 4 cm in diameter and 5cm deep) were sampled and composited, providing the base material with which each treatment was blended. Soil material was sieved with a 2 cm mesh to remove large rock fragments.

Field moist soil material was blended with lime and fertilizer, composted poultry manure or poultry manure and paper mill sludge (at produced moisture levels) as per quantities listed in Table 3-1 for a total of six different treatments. Proportions of each material were based on a dry weight total of 150g of material per experimental unit, but were mixed using moist material weights. Materials were stored at 4 °C for 2d after collection and mixed 1d prior to the start of the incubation. Wet materials were mixed inside plastic bags to homogenize material. A subsample of each treatment was dried at 105 °C for 48 hours and subsequently ground. Total N and C were calculated based on the % solids and % N and C measured by combustion on a Fisons NA 1500 Elemental Analyzer (Nelson and Sommers, 1996).

Year two soil samples

Soils for the year two incubation experiment were collected from pre-established research plots at the same AML site in Schuylkill County, PA as the year one soil samples. See chapter IV for further description of the field study. The field research plots had been in place for exactly one year and were composed of the same ratio of materials as the year one lab incubation (Table 3-1). Field plots measured 9.1 by 6.1 m and had five treatments replicated four times each. Five cores from within each plot were composited in the field. Immediately upon returning to the lab, samples were sieved using a 2 cm mesh sieve to remove large rocks and plant material while attempting to minimize destruction of all soil aggregates. Samples were stored at field moisture content at 4 °C for 2 d after collection and homogenized by mixing in plastic bags 1 d prior to the start of the incubation. Total N and C were measured using a CEInstruments Elemental Analyzer 1100 (Thermo Electron Corp) using oven dry (105 °C for 48 hours), ground subsamples.

Table 3-1. Amendment application rates added to mine soil for each experimental unit in the year one incubation experiment. A total of 150 g dry weight was added to each cup. Associated C and N added with each treatment are also listed.

Treatment		Composition	
	Material	C	N
	g	g kg^{-1}	mg kg^{-1}
Lime+fertilizer			
limestone	8.82	7.6	—
NH_4NO_3	0.25	—	630
TSP	0.79	—	—
KCl	0.29	—	—
soil	139.9	40.3	292
Compost 1			
compost	14.9	38.1	3000
soil	135.1	40.3	292
Compost 2			
compost	27.2	76.4	6000
soil	122.8	40.3	292
Manure+PMS 20:1			
layer manure	8.80	22.0	3000
paper mill sludge	17.7	37.6	—
soil	123.5	40.3	292
Manure+PMS 30:1			
layer manure	15.3	44.1	3000
paper mill sludge	27.5	67.2	—
soil	107.2	40.3	292

Laboratory incubations

To separate stable and labile N pools, a long-term incubation approach of controlled conditions and repeated leaching was used (Stanford and Smith, 1972; Keeney, 1982; Kaye et al., 2003). This method allows for the measurement of inorganic N potentially available for plant uptake or loss by leaching while simultaneously assessing the microbially relevant C, an important component of ecosystem functions (Robertson et al., 1999).

For both experiments Buchner funnel cups (90 mm diameter by 50 mm tall) were used as the incubation vessels. The bottom of each funnel was lined with a 1.2 µm glass fiber filter (VWR 696), a 3 µm glass fiber filter (Whatman GF/D) and approximately 3 cm of glass wool followed by 150 g dry weight equivalent of the various mine soil amendment mixtures. An additional glass fiber filter (Whatman GF/A) was placed on top of the soil to prevent any soil dispersion (Motavalli et al., 1995). The inner sides of a petri dish lid (92 mm diameter) were coated with vacuum grease and fitted to the bottom of each cup to prevent any water from leaving the cup. All cups were placed in storage bins containing small cups of deionized water to sustain humidity. The storage bins were kept in a temperature controlled room (27 °C) with the storage bin lids slightly open to prevent development of anaerobic conditions (Bremner and Douglas, 1971). Deionized water was periodically sprayed in a fine mist over the cups to add moisture between leaching events.

Incubated soils from both experiments were leached at 1, 6, 13, 22, 34, 49, 64, 80, 99, 118, 139, 169, 201, 230, 265, 299, 334, 365 and 392 d after the mixing of soil treatments. Following the method of Nadelhoffer (1990), soils were leached with a nutrient-extractant solution containing essential nutrients but no N. At 5 min intervals, a 200 ml aliquot of nutrient solution was added to each cup of soil and left to equilibrate for one hour before attaching the cup to a funnel with a rubber stopper fitted in a 250 ml side-arm flask. Water was drawn through the soil with a vacuum (-50 kPa) attached to the flask until water ceased to drip, usually < 5 min. An average of 85% of added nutrient solution was recovered. Soil surface filters disintegrated after repeated leaching events and were replaced after every five leaching events.

Collected leachate was frozen and later analyzed for inorganic N (NH_4^+ and NO_3^-) by flow injection colorimetry (Lachat Quickchem FIA+8000, 2003a, 2003b) and for total C and organic C by combustion (Shimadzu Analyzer TOC 5000-A). At the conclusion of the experiment (392 d), a 10 g (dry weight) subsample of soil was extracted with 50 ml of 0.5 M K_2SO_4 to remove any unleached inorganic N (Motavalli et al., 1995). Concentrations were converted to mg kg^{-1} soil by using leachate volume and initial dry mass. Labile N was defined as the sum of inorganic N ($NH_4^+ + NO_3^-$) leached and the inorganic N extracted with K_2SO_4. Stable N was calculated by subtracting labile N from initial soil N (Kaye et al., 2002b).

Microbial biomass was measured for all soils at the end of the experiment using the chloroform fumigation extraction method (Brooks et al., 1985). A 10 g (dry weight) subsample was placed in a chamber with a $CHCl_3$ atmosphere under vacuum (-70 kPa) for 5 d. After fumigation, soils were extracted (50 ml 0.5 M K_2SO_4) and total N measured using in-flow digestion and flow injection colorimetry on a Lachat Quickchem analyzer (2003c). Microbial biomass was calculated by subtracting total extracted N measured after fumigation from total N extracted before fumigation; this number was then divided by 0.69 to account for biomass not released via fumigation (Brooks et al., 1985).

Soil respiration was measured 1 d prior to each leaching event by sealing each soil cup in a 1 L wide-mouth plastic jar (Nalgene) and measuring CO_2 accumulated during a 6 h period. For treatments containing fresh manure, 5 L plastic jars were necessary to prevent CO_2 saturation. All jars were fitted with septa for gas sample extraction and the inner rims of jar lids were coated with vacuum grease to ensure a tight seal. Prior to sealing the jars, ambient air was thoroughly homogenized by fanning and an initial measurement of CO_2 was taken. After six hours, headspace in each jar was mixed and a 1 ml sample was extracted for immediate measurement using a LI-COR CO_2/ H_2O Analyzer (LI- 7000). Headspace concentrations of CO_2 were converted to mg C kg^{-1} soil by using atmospheric pressure, air temperature, jar volume, subsample volume, and oven-dry mass of soil (Kaye et al., 2002b; Robertson et al., 1999). Labile C was calculated by adding the total C lost by leaching and the C lost by respiration. Stable C was determined by subtracting initial C from labile C.

Statistical differences were assessed using analysis of variance (ANOVA); mean comparisons were determined with Fischer's protected least significant difference (LSD) (SAS Institute, 2003). A significance level of $\alpha = 0.10$ was chosen to protect against a Type II error: failing to observe a difference when in fact there is one. In this experiment, this type of error was deemed more problematic in the sense that a conclusion of no treatment differences could lead to the erroneous belief that N mineralization potential is the same for organic and inorganic treatments, regardless of application rates, for example. Lastly, simple linear regression was used to compare rates of added N and C to stable N and C pools.

Results and Discussion

Year one experiment

Nitrogen

The year one experiment was marked by rapid losses of NH_4^+ + NO_3^- (labile N) in the first few weeks of incubation, with much smaller inorganic N leaching losses thereafter (Fig. 3-1). The leaching losses can be divided into three distinct periods based on leaching trends over the course of the experiment: 0 – 66 d, 67 – 250 d and 251 – 392 d (Table 3-2). In the NH_4^+ fraction, the bulk of losses occurred immediately and declined to extremely low levels by day 66. Both manure+PMS treatments and the lime+fertilizer treatment lost large quantities of N in the form of NH_4^+ during the first period, whereas the compost treatments lost only a small quantity of NH_4^+ in comparison. By contrast, the largest amount of NO_3^- leaching during the first period occurred with the compost and lime+fertilizer treatments. These three treatments leached 30 to 40 times more NO_3^- than the manure+PMS treatments. A small pulse of NO_3^- was leached from the reference soil in the first period, suggesting accelerated mineralization of native soil N brought about by soil disturbance during sample collection and preparation. Robertson et al. (1988), in a 12 wk incubation study of a sandy clay loam soil fertilized with $Ca(NO_3)_2$, attributed a large portion of initial flush of mineralization to disturbance incurred in removing soil cores from the field and sieving them in the lab.

Ammonium losses were far smaller during the second period than the first, with the manure+PMS 30:1 treatment leaching slightly more NH_4^+ than the other treatments (Table 3-2). With NO_3^- leaching there was a reversal of treatment effects in the second period compared to the first period. During the second period, NO_3^- leaching from the manure+PMS treatments increased substantially and was three times greater than that from the compost and lime+fertilizer treatments. By contrast, these treatments showed a substantial decrease in NO_3^- leaching from all treatments. Nitrate leaching losses in the third period were much smaller than in the first two periods. The treatment differences were also smaller than in the first two periods, though the manure+PMS treatments still lost twice as much NO_3^- as the compost treatments.

These three periods demonstrate labile N pools of differing recalcitrance. During period one, readily available N was quickly mineralized until this pool was consumed, wherein inorganic N leaching losses approached zero. After day 66, a more recalcitrant pool of N began to mineralize and was largely depleted by day 292; thereafter, more recalcitrant N was slowly mineralized (Fig. 1, Table 2). The lower rates of N loss from compost treatments during the second and third period suggest a larger pool of more recalcitrant N that slowly mineralized throughout the experiment. Results of a 146 d incubation of poultry broiler manure conducted by Gordillo and Cabrera (1997) concluded that N mineralization could be divided into a fast pool and a slow pool using a first-order kinetics model. Therefore, the N pools of differing recalcitrance observed in this experiment seem reasonable.

Interestingly, although there were distinct differences in quantities and timing of NH_4^+ or NO_3^- lost by leaching over the course of the year, the total leaching loss of labile N in the first year was not measurably influenced by the form in which N was added with these treatments (Table 3-3). These results suggest that under the intense leaching regimen of the incubation, all treatments, whether inorganic or organic, have the same potential for N mineralization and subsequent loss of inorganic N to leaching.

When compared to levels of N in soils at the start of the experiment, however, the organic treatments lost 16 to 27% of original N (Table 3-3). The amendment with the most initial N, compost 2, lost only 16% of initial N, compared to compost 1, which lost 21% of initial N (Table 3-3). Despite starting with 1.6x more N than compost 1, compost 2 still lost less N, confirming the ability of compost to minimize N leaching with large application rates. Both manure+PMS treatments lost the same amount of N as compost 1 during the one year experiment, demonstrating the potential of fresh manure mixed with PMS to minimize leaching losses as efficiently as compost containing the same quantity of N. Conversely, the lime+fertilizer treatment lost 68% of N originally added, suggesting a substantial exhaustion of the N pool in this treatment. Losses of this magnitude were also measured by Cabrera et al. (1993) in a 35 d incubation study with pelletized poultry manure.

Stable N pools in the organic amendments were substantial at the end of the first year (Table 3-3). As expected, the higher rate of compost application produced the largest N pool in concordance with the elevated N addition. What was more notable, however, was the lack of difference in stable N pool size between compost 1 and both manure+PMS treatments. These results reveal that, when initial N was the same, manure+PMS was just as effective as compost at increasing the stable N pool. Moreover, this increase in the stable N pool was achieved with a C:N ratio of 20:1, meaning the additional C applied with the 30:1 treatment was unnecessary to both minimize leaching and increase stable N.

These results are also consistent with studies involving the addition of ^{15}N tracers to forest soils in order to monitor labile and stable pools, which have shown that stable pools are much larger than labile pools, even after intense leaching schedules. In these studies, however, quantities of N added were typically < 4% of that applied with any of the organic treatments here. In a one year lab incubation study of plantation soils from Puerto Rico, Kaye et al. (2002a) reported that more than two thirds of added N was retained. Likewise, Tietama et al. (1998) and Nadelhoffer et al. (1999), in two separate field studies involving coniferous and temperate forest soils, respectively, measured approximately 75 to 80% retention of added N in soils with ^{15}N tracer additions. Furthermore, these studies noted a direct correlation between N added and stable N pools; this was also the case in my study ($r^2 = 0.98$; $p = 0.0661$), suggesting that even with large N additions, the soil N retention capacity of these mine soils was not saturated.

The much smaller stable N pool generated by the lime+fertilizer treatment has implications for the restoration of soil productivity and sustainability of nutrient cycling in these nutrient poor mine soils and the long-term development of a stable plant community. Based on these results, lime+fertilizer would not be a good choice for retaining N and improving ecosystem function. Interestingly, the reference soil stable N pool was larger than that of the lime+fertilizer treatment, suggesting that, under the intense leaching conditions of this incubation, the addition of fertilizer caused the stable N pool to be smaller than if no amendment had been added to the soil. Marinari et al. (2000) also observed this “priming effect” in a field experiment involving the application of NH_4NO_3 (200 kg N ha^{-1}) to a sandy clay loam soil. They concluded that the large addition of N primed native soil organic matter, providing a nutrient source for the microbial biomass and resulting in enhanced CO_2 production and mineralization of N.

Microbial biomass, a fraction of the stable N pool, was two times larger in the manure+PMS 30:1 treatment than the manure+PMS 20:1 treatment (Table 3). Compared to the compost treatments, four to eight times more microbial biomass was measured in the manure+PMS treatments. The significantly larger microbial pools in the manure+PMS treatments suggests more microbially available C may remain after 1 yr of intensive leaching compared to compost treatments. A 360 d incubation study by Pascual et al. (1999) demonstrated long-term soil benefits of adding fresh sewage sludge to soils as opposed to composted sludge, wherein properties such as soil organic matter and microbial biomass were measurably greater in soils amended with fresh material even after 8 yr. Therefore the addition of manure+PMS, as opposed to compost, could have substantial impacts on the sustainability and long-term nutrient cycling in these soils.

Although the compost treatments had less microbial biomass than the manure+PMS treatments, they still had considerably larger pools than the lime+fertilizer and reference soils (3x to 4x larger). The difference between microbial biomass in organic treatments compared to the lime+fertilizer treatment was not surprising given the large and small pools of stable N and C associated with the organic and inorganic treatments, respectively. In addition, microbial biomass was not different between the lime+fertilizer and reference soil, signifying that there has been no substantial increase in the microbial pool with the lime+fertilizer treatment.

The small size of the microbial pool relative to the total stable N pool, however, shows that microbes are not directly responsible for storing all of this non-labile N in their biomass; rather, microbial N turnover is more likely the mechanism for N retention, a hypothesis proposed and by Stark and Hart (1997) and confirmed by Kaye et al.

(2002b). This turnover appears to be essential for maintaining large soil N pools that are capable of providing N for plants and minimizing N leaching.

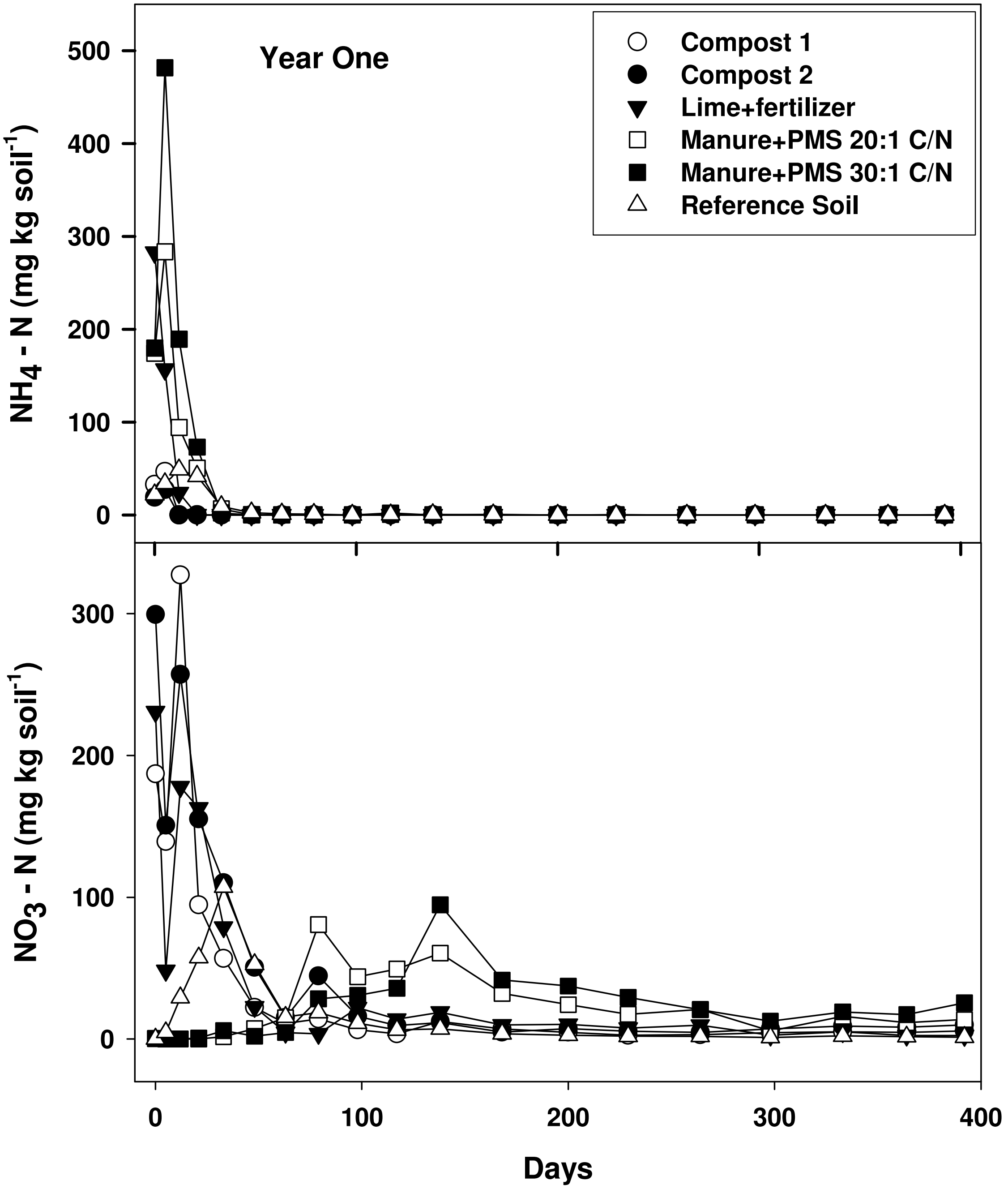

Figure 3-1. NH_4^+ and NO_3^- leached during the year one incubation (0 – 392 days).

Table 3-2. Leached NH_4^+, NO_3^- and labile N ($NO_3^- + NH_4^+$) for incubated one year soils (days 0 – 392) and total N leached from year two (days 365 – 757). Total refers to N leached plus N measured by a K_2SO_4 extraction. For each analyte values within columns followed by the same letter are not significantly different at $\alpha = 0.10$.

	Year One					Year Two
	Period			K_2SO_4	Total	Total
	0 – 66d	67 – 250d	251 – 392d	0 – 392d	0 – 392d	365 – 757d
	mg N kg soil^{-1}					
NH_4^+						
Lime+fertilizer	464b	0.596b	0.187a	0.266b	465b	5.50a
Compost 1	79.6c	0.406b	0.345a	0.559b	80.9c	4.94a
Compost 2	47.0c	0.425b	0.148a	1.07b	48.6c	5.36a
Manure+PMS 20:1 C/N	609b	0.743b	0.00a	2.11a	612b	4.67a
Manure+PMS 30:1 C/N	926a	3.09a	0.014a	2.91a	932a	4.06a
Reference Soil	159c	2.06ab	0.092a	0.563b	162c	—
NO_3^-						
Lime+fertilizer	726b	46.6c	9.95c	7.07d	790b	216cd
Compost 1	838ab	78.6bc	15.6c	20.5cd	953b	308bc
Compost 2	1038a	144b	27.3bc	27.3bc	1240a	131d
Manure+PMS 20:1 C/N	24.5c	335a	41.3ab	36.7b	438c	393ab
Manure+PMS 30:1 C/N	13.7c	346a	62.0a	81.2a	503c	413a
Reference Soil	267c	55.0c	5.09c	8.24d	335c	—
Labile N						
Lime+fertilizer	1190a	47.2c	10.1c	7.34d	1260a	221cd
Compost 1	918ab	79.0bc	15.9c	21.1cd	1030a	313bc
Compost 2	1090a	144b	27.4bc	28.4bc	1290a	136d
Manure+PMS 20:1 C/N	634bc	336a	41.3ab	38.8b	1050a	397ab
Manure+PMS 30:1 C/N	940ab	349a	62.0a	84.2a	1440a	417a
Reference Soil	426c	57.1c	5.18c	8.80d	497b	—

Table 3-3. Labile and stable N pools by treatment for year one and year two. Labile N was calculated by adding labile N (NH_4^+ + NO_3^-) leaching losses and K_2SO_4 extracted labile N. Stable N pool was determined by subtracting labile N from N added. Microbial biomass constitutes part of the stable N pool. Within rows values followed by the same letters are not significantly different α = 0.10.

	Treatments					
	Lime+fertilizer	Compost 1	Compost 2	Manure+PMS 20:1 C/N	Manure+PMS 30:1 C/N	Reference soil
	mg N kg^{-1}					
Year One						
Starting N	1824	4944	7944	4944	4944	1944
Leaching (NH_4^+ + NO_3^-)	1247a	1013a	1257a	1011a	1351a	489b
N loss (%)	68.3	20.5	15.8	20.4	27.3	25.2
K_2SO_4 Soil Extraction (NH_4^+ + NO_3^-)	7.34d	21.1cd	28.4bc	38.8b	84.2a	8.80d
Microbial Biomass (Total N)	4.46c	20.5c	15.0c	81.7b	167a	9.05c
Labile N	1250a	1030a	1290a	1050a	1440a	498b
Stable N	574d	3910b	6650a	3890b	3500b	1450c
Year Two						
Starting N	1632	5455	9576	4463	4739	1944
Leaching (NH_4^+ + NO_3^-)	120d	271c	300bc	382ab	400a	489a
N loss (%)	7.35	3.99	3.13	8.55	8.42	25.2
K_2SO_4 Soil Extraction (NH_4^+ + NO_3^-)	3.74b	13.1a	16.0a	15.6a	17.7a	8.80ab
Microbial Biomass (Total N)	269b	788a	913a	929a	816a	9.05c
Labile N	121e	274cd	312bc	386ab	405a	498a
Stable N	1511c	5181b	9264a	4077b	4334b	1450c

Carbon

Loss of C by leaching was not a significant loss pathway in this experiment, since with all treatments such losses were a small fraction of initial C (Table 3-4). Most C leaching occurred in the first 14 d of incubation, with very little C leached from all treatments thereafter (Fig. 3-2). The manure+PMS treatments lost nearly 10 times more C by leaching than did the compost treatments which in turn lost only slightly more C than the lime+fertilizer and reference treatments. Labile C is greatly reduced in the process of composting, which would account for the large differences in C leached between manure+PMS and compost treatments, despite similarly large initial C inputs (Hanselman et al., 2004).

CO_2 fluxes measured the respiration potential of treated soils and accounted for almost the entire labile C pool in these systems (Table 3-4). Respiration rates for the manure+PMS treatments peaked on day 14, declined over the next 124 days, and stabilized at relatively low levels for the duration of the experiment (Fig. 3-3). Respiration losses from these treatments were greater than all other treatments during the first 92 days of the experiment; compost treatments did not exhibit an elevated rate of C loss by respiration during that same period. Bernal et al. (1998), in an incubation study of a silt loam soil mixed with manures at various stages of composting, also reported that C respiration rates were highest from fresh, unstabilized manures; these losses were decreased through stabilization, as was observed with compost treatments in my experiment. Both manure+PMS treatments lost 36% of initial C by respiration regardless of C:N ratio. These higher respiration rates also point to the existence of easily mineralizable C, meaning that the quality of C in the PMS is not so recalcitrant as to be immediately unavailable to microbes (Bernal et al., 1998; Paré et al., 1998).

Compost treatments lost 5 – 7% of initial C through respiration despite initial C that was 1.5 to 2.5 times greater than the lime+fertilizer treatment. Flavel and Murphy (2006), in a 142 d incubation study, found similar percent losses of C as CO_2 for both green waste and straw based compost treatments mixed in a coarse textured soil. Mineralization of labile C during the composting process would account for the relatively small respiration losses from compost treatments once applied to soil (Amlinger et al., 2003). Approximately 46 to 62% of C was lost in the process of composting beef manure in an incubation study by Eghball et al. (1997). Assuming C losses of a similar magnitude in production of the compost in my experiment, the *in situ* "composting" of the manure+PMS treatments would have resulted in slightly smaller respiration losses of the organic C in the organic materials. Lime+fertilizer and reference soil respiration losses did not differ and were significantly lower than respiration rates of organic treatments, representing approximately 6% of initial C and suggesting that microbial populations were less active in these treatments.

Unlike stable N, stable C pools did not correlate as well with quantities of added organic C. The compost 2 treatment again had the largest stable C pool, but started with 22% less C than the manure+PMS 30:1 treatment. The manure+PMS 20:1 treatment, despite having 1.3x more initial C than the compost 1 treatment, had a smaller pool of stable C at the end of the incubation. Additionally, the lime+fertilizer treatment had a larger stable C pool than the reference soil. In the case of stable C, C:N ratio appears to play a greater role for the manure+PMS treatments than it did for stable N.

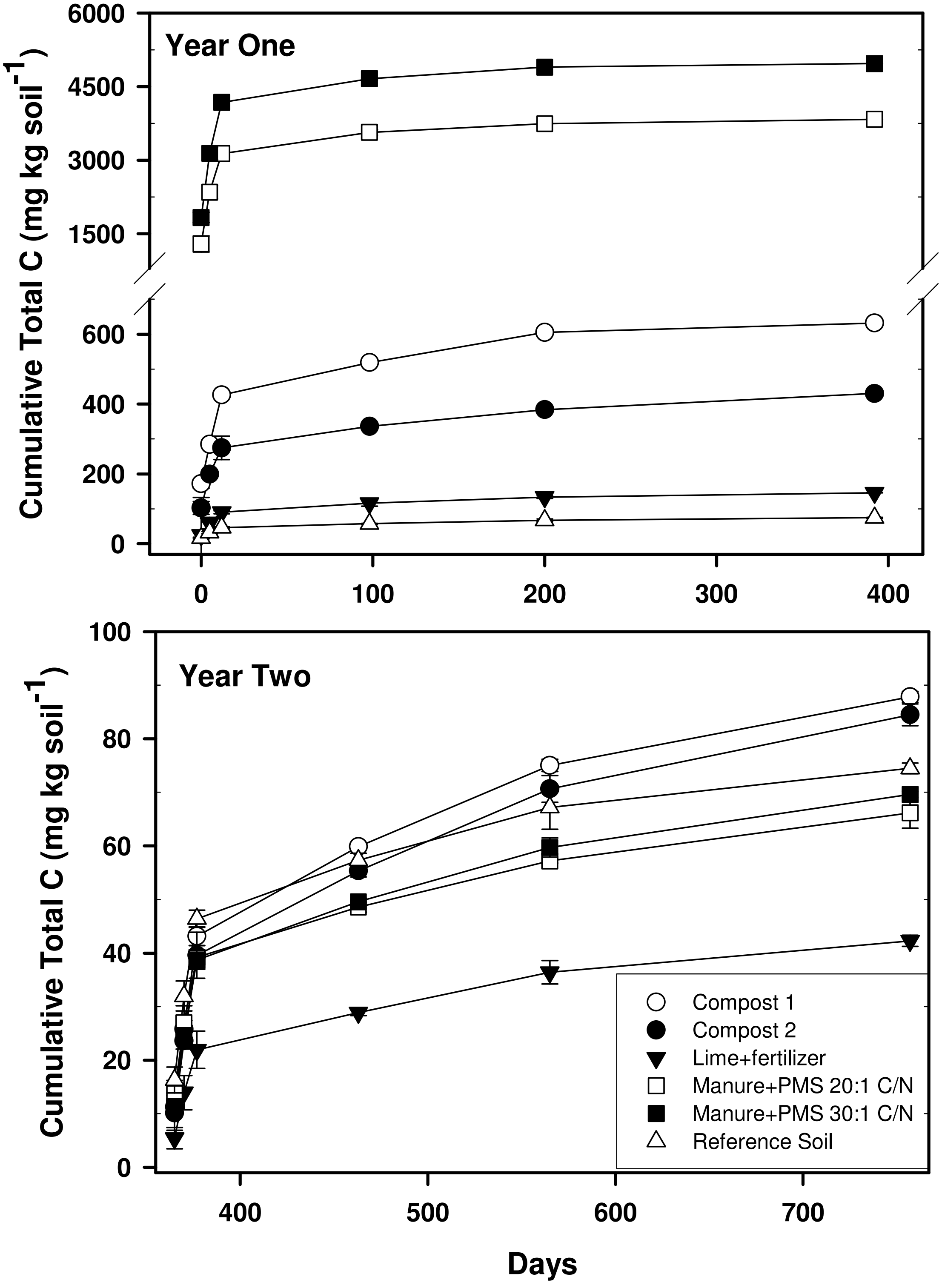

Figure 3-2. Cumulative total C leached during year one (top) and year two (bottom) incubations. Note difference in scale between the two experiments.

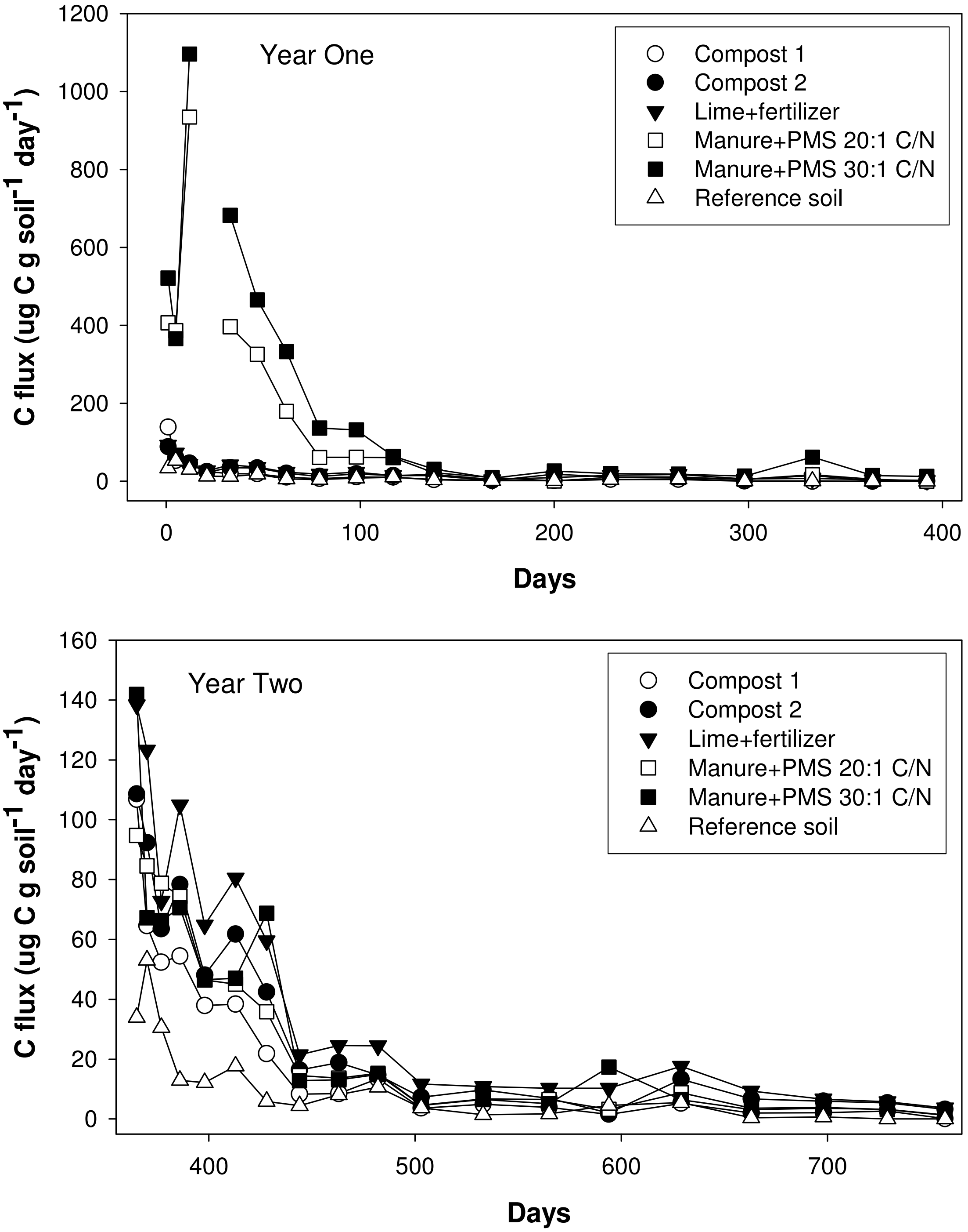

Figure 2-3. C fluxes for year 1 and year 2 incubations. Note scale difference between plots. Analytical problems on day 21 of the year one experiment resulted in no data for the Manure+PMS 20:1 and 30:1 treatments.

Table 3-4. Labile and stable C pools by treatment for year one and year two. Labile C is the sum of leaching and respiration losses. The stable C pool was determined by subtracting labile C from initial C. Within rows values followed by the same letter are not significantly different at $\alpha = 0.10$.

	Treatments					
	Lime+fertilizer	Compost 1	Compost 2	Manure+PMS 20:1 C/N	Manure+PMS 30:1 C/N	Reference soil
	g C kg^{-1}					
Year One						
Initial C	47.9	78.4	117	99.9	151	40.3
Leaching losses	0.123d	0.406cd	0.574c	1.89b	3.33a	0.0662d
Respiration losses	2.98d	5.66c	6.25c	36.1b	54.6a	2.28d
Respiration C loss (%)	6.22	7.22	5.34	36.1	36.2	5.66
Labile C	3.10c	6.07c	6.82c	38.0b	57.9a	2.35c
Stable C	44.8e	72.3c	110a	62.0d	93.1b	38.0f
Year Two						
Initial C	43.9	86.0	120	86.3	132	40.3
Leaching losses	0.0478c	0.0802a	0.0784a	0.0635b	0.0630b	0.0662b
Respiration losses	4.46c	7.18b	9.76a	6.14b	6.80b	2.28c
Respiration C loss (%)	10.2	8.35	8.13	7.11	5.15	5.66
Labile C	4.51c	7.26b	9.83a	6.20b	6.86b	2.35d
Stable C	39.4d	78.7c	110b	80.1c	125a	38.0e

Year two experiment

Nitrogen

N leaching losses from all treatments in the year two experiment (360 day incubation of soils collected one year after amendment application in the field) were much lower than N losses in the year one incubation (Table 3-2). There were no discernible trends in leaching of labile N (either fraction) over time in the two year experiment. Overall, quantities of NH_4^+ lost from all treatments were similar to that lost in the second period of the year one experiment, a period associated with more recalcitrant N in the previous incubation. Likewise, NO_3^- losses in year two were close to values measured in period two of year one.

The lack of readily mineralizable N in the second year incubation implies that in the field, the potential labile N loss suggested by the year one experiment did not occur in the field; rather it was turned over via microbial biomass and plant uptake and was transformed into more recalcitrant forms. Starting N values for year two treatments were higher than year one for the two compost treatments, meaning that these treatments generated an even larger stable N pool than was predicted by the year one incubation (Table 3-3). Additionally, this advocates that plants, in addition to microbes, were important in cycling and transforming N into more recalcitrant forms, given their absence from the incubations and presence in the field. Even the lime+fertilizer treatment has created a stable N pool 3x larger than predicted, suggesting that far less N was mineralized in the first year in the field than anticipated.

Although leaching losses were small compared to values measured in year one, there were some treatment differences in cumulative labile N leached in year two (Table

3-2). Manure+PMS treatments leached slightly more N than compost treatments, accounting for approximately 8% of initial N and a larger reduction in percent loss compared to year one. Compost treatments only lost 3-4% of initial N, again a smaller fraction of initial N than was lost in the first year. The lime+fertilizer treatment leached the lowest amount of labile N of all treatments in year two, but given the lower starting N, this still constituted almost 8% of initial N.

Stable N pools at the end of the incubation, however, showed the same trend as the one year incubation (Table 3-3). As expected, compost 2, with the highest starting N, had the largest stable N pool, validating the hypothesis that compost is an extremely effective way to add large quantities of stable N to the soil. Once again, however, manure+PMS treatments, regardless of C:N ratio, generated stable N pools as large as compost 1, even though the starting N values were not the same in the second year. These results support that manure+PMS treatments were just as effective at incorporating N into a stable N pool as compost treatments. Additionally, the extra C added with the 30:1 ratio was unnecessary to achieve a large stable N pool in the year two incubation.

By contrast, the stable N pool associated with the lime+fertilizer treatment was substantially smaller than all organic treatments. Not only was this treatment relatively ineffective at building a large stable N pool in both years of incubation, it is unlikely the fertilizer N will be retained in the long-term. An eight year field study by Preston and Mead (1994), in which pine trees were fertilized at a rate similar to that used in the lime+fertilizer treatment, found that approximately 50% of the N that had been retained after the first year was no longer in the soil N pool 8 yr later.

Microbial biomass pools were much larger in year two than in year one for all treatments (Table 3-3). Contrary to results of the year one incubation, there was no difference in microbial pool size among organic treatments, indicating that larger pools were generated with all treatments during the year in the field. The pool size of the lime+fertilizer treatment, however, was not significantly different than the reference soil, further implying that this treatment does little to improve soil quality in terms of microbial activity.

Carbon

Leached C represented an even smaller fraction of the labile C pool in the year two experiment than it did in the year one experiment, but followed the same trend of largest leaching rates during the first 14 d and small rates of loss for the duration of the incubation (Fig. 3-2). Unlike the year one experiment, manure+PMS treatments leached less total C than compost treatments (Table 3-4).

CO_2 flux rates during the first 99 d of the experiment showed no clear treatment effects and were likely an artifact of soil disturbance (Fig. 3-3). Flux rates for manure+PMS were an order of magnitude smaller than fluxes recorded in the year one experiment and were also less than the C flux from the compost 2 treatment (Table 3-4). These low respiration rates from the manure+PMS treatments point to a more stabilized compost-like product after one year of reaction in the field (Bernal et al., 1998). Although respiration losses were lowest from the lime+fertilizer treatment, this loss accounted for 10% of initial C, a higher percentage than all other treatments. In a 3 d incubation of soils collected from reclaimed mine sites in Wyoming, Ingram et al. (2005)

also found that microbial respiration was much lower from mine spoil than from mine soils that had been reclaimed using traditional lime and fertilizer, implying that despite low respiration rates, the lime+fertilizer treatment still had more microbial activity, and presumably more nutrient cycling, than unamended mine soil.

Similar to starting N values, initial C values also differed from the year one incubation. The compost treatments started with slightly more C in the second year, as did the manure+PMS treatments. The lime+fertilizer treatment, however, actually started the year two experiment with slightly less C than the year one, which is likely a result of less precision and accuracy in applying amendments in the field. Considering that these soils were in field conditions for one year, biomass addition to the soil likely added C to the soil. Even with these additions, however, the lime+fertilizer treatment started the experiment with less C than expected, signifying that this treatment was not very effective at stabilizing C.

Stable C pools at the end of the experiment were directly proportional to the starting quantity of C, with the manure+PMS 30:1 and compost 2 treatments generating the largest stable C pools. Furthermore, stable C pools did not differ between the manure+PMS 20:1 and compost 1 treatments. Given that these treatments started the year two incubation with almost exactly the same amount of C, both treatments, regardless of C source, were equally effective at building a large stable C pool. Compost, having gone through the initial stabilization of N and C during the production of the material, showed more consistent C pools between year one and year two, as would be expected. Given the large retention rates of added N even after two years, these large pools of stable C should provide a substantial long-term sink for the added N (Kaye et al., 2002a).

Conclusions

Both the year one and year two lab incubation experiments were successful at differentiating the labile and stable N and C pools associated with various treatments. In both years, organic treatments were substantially more effective at building large stable N and C pools, regardless of whether manure was composted or applied in conjunction with paper mill sludge. As expected, compost treatments, and especially the higher rate of compost addition (compost 2), were very effective at minimizing inorganic N leaching losses and building large N and C pools proportional to the quantity of N and C added with these treatments. Given that these incubations involved more intensive leaching conditions than ever would occur in a natural setting, these results confirm that compost treatments are superb at retaining N and C in stable pools and promoting nutrient cycling, as evidenced by large microbial biomass pools.

More notable, however, was the fact that manure+PMS treatments, regardless of C:N ratio, were just as effective as compost at minimizing N leaching, building large stable N and C pools, and generating large microbial pools. Additionally, there appears to be more microbial activity associated with these treatments than with compost treatments, which could translate to enhanced, long-term soil nutrient cycling. When considering C and potential N losses incurred in the process of composting manure, applying manure+PMS directly to the soil may actually result in less N and C loss from the original organic materials, thus maximizing the potential sequestration of C and N from those materials. Finally, there was apparently no benefit from increasing C:N ratio from 20:1 to 30:1 by adding more PMS since the N leaching losses and stable N pools were not measurably different.

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Chapter IV

Mine reclamation with poultry manure: effects on nitrogen leaching, soil carbon and switchgrass growth

Introduction

Field scale studies are important in understanding how results observed in controlled greenhouse or lab incubations translate to conditions of the natural environment, which are far more unpredictable and variable, but nonetheless more accurately reflect reality. The greenhouse study presented in Chapter II demonstrated that poultry manure could be used for mine soils reclamation to ameliorate soil phytotoxicity and improve switchgrass growth. The greenhouse experiment also demonstrated that using composted manure or mixing manure with a high C, low N material, leaching of N and soil N sequestration could be greatly increased. However, the experiment was performed under controlled environmental conditions, was very short in duration (8 mo), and involved an intense leaching schedule and an unsustainable switchgrass harvest schedule. Thus, while the greenhouse experiment served as a proof of concept for manure use in reclamation, it did not demonstrate that these amendments would be similarly effective under natural conditions and stresses. Therefore it was essential that these concepts be tested in a long term field experiment.

Field studies reported in the literature have shown poultry manure can be used to improve soil nutrients and plant growth. Composted manure has been shown to be extremely effective at minimizing N losses and improving vegetative growth (relative to fresh manure) and several studies have demonstrated that even with large compost application rates, quantities of nitrate in soil and leachate are low compared to fresh poultry manure or inorganic fertilizer amended soils (Cooperband et al., 2002; Hartl et al., 2003). Field experiments also allow investigation of application methods and yield responses. In a study by Eghball and Gilley (1999), higher concentrations of runoff N and P were observed with both compost and fresh cattle manure applications to a silty clay loam soil when amendments were under no-till conditions rather than disked into the soil, indicating that incorporation is an important factor in retaining nutrients.

Other field studies involving biosolids have helped understand the seasonal patterns of N loss. Stehouwer et al. (2006), in a field study of biosolids application to abandoned mine soils, observed pulses of NO_3^- leached in the fall two consecutive years after application. Daniels et al. (2001), however, observed a one-time pulse of NO_3^- leached in the fall following amendment application. Such timing of N losses must be assessed in the field, where precipitation and climate are not controlled and could have important implications for developing reclamation practices that would minimize nutrient losses. A field study by Schmidt et al. (2001) also demonstrated the effectiveness of the co-application of biosolids with sawdust to reduce N leaching, suggesting that results of the greenhouse experiment, which suggested C:N ratio adjustment was indeed a viable option for stabilizing manure N, will potentially work just as well under field conditions.

To understand how composted or fresh manure mixed with paper mill sludge treatments behave under natural conditions, a field study was designed on an abandoned mine soil in Schuylkill County, PA. Based on the results of the greenhouse study, application rates were selected to test in the field (chapter II). Because I observed a distinct linear response in switchgrass growth with increasing N application rates and effective reduction of N leaching by composting and C:N ratio adjustment, I decided to use larger N application rates in the field experiment. Although the 40:1 C:N ratio mix produced the largest switchgrass yield in the greenhouse study, it had a negative environmental effect of increased N leaching and therefore was not considered for further testing the field. The 30:1 C:N adjustment produced higher yields and reduced N losses compared to the 20:1 C:N, but the lower ratio was still of interest because this treatment requires less material to be hauled, spread and incorporated, which means a lower cost of implementation.

The objectives of the field experiment were to determine the effectiveness of composting manure and C:N ratio adjustment of manure for:

1. Controlling nutrient leaching when used for mine reclamation,
2. Enhancing soil sequestration of nitrogen and carbon, and
3. Increasing productivity of switchgrass grown on reclaimed mine soil.

Materials and Methods

A multi-year field experiment was initiated in the spring of 2006 at an abandoned surface coal mine in Schuylkill County, Pennsylvania, within the anthracite coal belt of eastern PA. The site was last mined in the 1950s and was reclaimed by backfilling with overburden material and a thin cover of fine earth material. Presently, the site is classified taxonomically as an Udorthent strip mine (NRCS, 2009). The site was nearly level (< 1% slope, SE – NW), although the northwest corner was often wetter than other parts of the site. Vegetative cover was moderate, which included grasses and some woody species. Initial site texture was a very channery sandy loam with approximately 43% rock fragments (mostly shale). Annual precipitation at this site averaged 1740 mm (46 inches). Bulk density was 1.77 g cm^{-3} and soil pH was 5.1 (1:1 in water), with an initial liming requirement of 8960 kg ha^{-1} (8000 lb ac^{-1}) for a pH of 6.5 (Eckert and Sims, 1995). Initial total soil carbon was measured at 3.1%, reflecting the presence of coal fragments in the mine soil (Nelson and Sommers, 1996).

Five reclamation treatments were each replicated four times in a randomized complete block design with each plot measuring 6.1 m by 9.1 m. The treatments included a standard reclamation practice of ground limestone and inorganic fertilizer (ammonium nitrate, triple super phosphate and KCl) amendment, two rates of composted poultry layer manure, and two blends of fresh poultry layer manure and paper mill sludge (PMS). Table 4-1 shows treatment quantities and compositions, along with their respective soil additions of C, N, P and K. These rates were chosen based on results obtained in the greenhouse study which demonstrated a significant linear response of switchgrass growth to increased applications of compost or manure+PMS (see chapter

II). These results, coupled with the low loss of N and P under an intensive leaching regimen, prompted even higher rates in the field study to further test the capacity of these treatments to retain nutrients and improve vegetative growth. The lowest rate of compost and the two manure plus paper mill sludge (manure+PMS) blends contained the same initial quantity of total N (almost all from manure); paper mill sludge was added to fresh manure to adjust the C:N ratio to 20:1 and 30:1 (manure+PMS mixes) (Table 4-1). Poultry manure had an initial C:N ratio of 7.3:1 while the paper mill sludge had a C:N ratio of 126:1. C and N were measured using the combustion method on a Fisons NA 1500 Elemental Analyzer (Nelson and Sommers, 1996). Initial pH of the manure and paper mill sludge was 8.3 and 7.3, respectively. Compost was manufactured by mixing poultry layer manure with leaves, shredded wood and water prior to forming an open windrow. During active composting the windrow was turned every 7 to 14 days depending on temperature for 30 d; compost was then matured for two months in a static pile. Compost, fresh manure and paper mill sludge were hauled to the experiment site and mixed on site to produce the desired C:N ratio blends. All amendments were surface applied and then incorporated into the upper 5 to 8 cm of the soil using the teeth on a front end loader bucket. Due to the extremely rocky nature of the site, it was not possible to utilize conventional tillage equipment or to achieve deeper incorporation.

All plots were initially planted with a combination of 11.2 kg ha^{-1} (10 lbs ac^{-1}) of switchgrass (*Panicum virgatum* L.) and 2.2 kg ha^{-1} (2 lbs ac^{-1}) of annual ryegrass (*Lolium rigidum* Gaud.) immediately following amendment application; the ryegrass was included as a nurse crop to provide some rapid cover prior to switchgrass establishment. Following seeding, one bale of straw mulch was applied to each block (five plots) to minimize soil erosion. Vigorous ryegrass growth prevented the establishment of switchgrass in the first year after planting, therefore plots were reseeded with 22.4 kg ha^{-1} (20 lbs ac^{-1}) of switchgrass seed in spring 2007. To minimize ryegrass competition in the second year, the plots were mowed at approximately 15 cm (6 in) in May and June of 2007 and June of 2008. No pesticides, herbicides, or additional fertilizer were applied to plots.

Table 4-1. Amendment application rates (dry weight basis) added to field plots and associated C, N, P and K added with each treatment. CCE refers to the quantity of alkalinity expressed as Mg of $CaCO_3$ ha^{-1} based on the CCE.

	Additions to minespoil					
Treatment	Material	CCE	C	N	P	K
	Mg ha^{-1}			kg ha^{-1}		
Lime+fertilizer						
Limestone	13.4	13.4	1.61	—	—	—
NH_4NO_3	0.148	—	—	112	—	—
TSP	0.452	—	—	—	196	—
KCl	0.166	—	—	—	—	186
Compost 1	78	20.9	27.0	2117	1052	81.3
Compost 2	156	41.9	54.1	4234	2104	163
20:1 manure+PMS						
layer manure	50	19.4	15.5	2117	1052	216
paper mill sludge	103	36.6	27.0	—	—	—
30:1 manure+PMS						
layer manure	50	19.4	15.5	2117	1052	216
paper mill sludge	184	65.6	48.2	—	—	—

Site instrumentation

Prior to amendment application all plots were instrumented with 30 cm by 30 cm zero-tension pan lysimeters 30 cm below the surface to collect vadose zone leachate. The rocky soil prevented lateral installation of the pan lysimeters below undisturbed soil, therefore trenches were dug to install lysimeters in the center of each plot. Although this disturbed the soil, the material had already undergone severe disturbance following mining activities, hence this disturbance did not greatly alter the soil profile. Some large rocks were removed from near the pans in an attempt to minimize the creation of large pores that would preferentially transport large volumes of water to the lysimeters. Polystyrene beads were used to fill pans and drain lines (approximately 3 m long) drained leachate collected by lysimeters to carboys located in dry wells outside the plot area. Excavated soil material from the upper 10 cm and lower 20 cm was kept separate during trench excavation and replaced in sequence. A rain gauge was installed in the northwest corner of the plot area and an electric fence was erected around plots to discourage grazing by resident wildlife.

Sample collection and analysis

Leachates were collected as soon as possible following all precipitation events large enough to generate lysimeter flow. Leachate volumes were measured in the field and a 500 ml sample was taken back to the lab for processing and analysis. Two 60 ml aliquots were filtered (0.45-µm membrane filter); one was acidified (0.25 ml concentrated HNO_3). Unfiltered samples were used to measure pH (potentiometric) and electrical conductivity (EC). Filtered samples were used in analyzing concentrations of

NO_3^-, NH_4^+, and PO_4^{-3} using flow injection colorimetry on a Lachat QuickChem FIA+8000 Analyzer; concentrations of total N and total P were measured on the same instrument using in-line digestion followed by flow injection colorimetry (Lachat Instruments, 2003a, 2003b, 2007a, 2003c, 2007b). Filtered and acidified samples from the first month of leachate collection were analyzed for Ca, Cd, Cr, Cu, Mg, Mo, Na, Ni, Pb and Zn (inductively coupled plasma emission spectrophotometry [ICP]) and the first nine months of leachate were analyzed for Ca, Mg, Na and K (ICP). Quantities of nutrients lost in each leaching event were calculated by multiplying nutrient concentrations by leachate volume collected in each lysimeter. Cumulative losses were determined by assuming the lysimeter collected leachate from a 30 by 30 cm area; these data were summed by year and over the entire 55 month collection period.

Biomass yield data were obtained by clipping all vegetation present in 1 m^2 quadrats randomly located within each plot in late summer of each year; weeds did not greatly influence biomass data. Harvested plant material was dried and weighed to determine biomass yield. Tissue samples from each year were analyzed for total N and C (combustion on an Elementar Vario Max N/C Analyzer), P, K, Ca, Mg, Al, B, Cu, Fe, Mn, Na, and Zn (dry ash method) (Horneck and Miller, 1998; Miller, 1998).

Surface soil samples were collected prior to amendment application, in spring and summer of 2006 and 2007, and in spring, summer, and fall of 2008. Five independent cores from each plot were extracted with a soil auger to a depth of 5 cm; the rocky soil prevented deeper sampling in 2006 and 2007. In 2008, soils were sufficiently workable to collect samples at both 0 – 5 and 5 – 10 cm depths. Cores were homogenized by plot and depth, ground, and analyzed for pH (1:1 in water), total N (combustion on a Fisons

NA 1500 Elemental Analyzer), total C (combustion on a CEInstruments Elemental Analyzer 1100), and Mehlich-3 extractable P, K, Ca and Mg (ICP) (Eckert and Sims, 1995; Bremner, 1996; Nelson and Sommers, 1996; Wolf and Beegle, 1995). Bulk density was measured in August 2008 using the excavation method (Blake and Hartage, 1986).

To test for statistical differences, an analysis of variance was calculated followed by Fischer's Protected Least Significant Difference (LSD) for mean comparisons. A significance level of $\alpha = 0.10$ was chosen to protect against a Type II error in analysis of leachate, soil and tissue concentrations. A Type II error in this experiment (failing to conclude that there was a difference in N leached, for example, when this was actually the case) would be more serious than the alternative given the environmental sensitivity associated with nutrients such as N and P. A significance level of $\alpha = 0.05$ was used for yield data where a Type I error (concluding a difference in yield when there was in fact no difference) was deemed more important to consider. Because treatment effects were likely to diminish over time and follow seasonal patterns, each year of data was analyzed separately. All statistical analysis was performed using SAS software (SAS Institute, 2003).

Results and Discussion

Variability was quite high for almost all measured parameters in this experiment. This variability likely came from the inherently heterogeneous nature of the mine soil itself, as well as non-uniform spreading and incorporation of amendments, resulting from the rocky soils. Furthermore, not all lysimeters operated at the same efficiency; consistently large volumes collected from some lysimeters suggested preferential flowpaths while the repeated absence of leachate in others implied flow diversion or clogged drain lines. Soil nutrient concentrations were also variable, again due to non-uniform incorporation of materials. Despite the large variability, I was able to determine significant treatment effects for many of the parameters measured in this experiment.

Nutrient leaching

Nitrogen

Throughout the three years of the experiment most of the N lost by leaching was inorganic, except for leachates from compost treatments in the first year (Table 4-2). Inorganic N losses were mostly as NO_3^-, although during the first two months after application more NH_4^+ than NO_3^- was lost from the manure+PMS treatments. The inorganic N (NH_4^+ + NO_3^-) losses from the manure+PMS treatments showed a temporal pattern, with large pulses in the fall of the first two years (Fig. 4-1). There was relatively little loss in the intervening months as leachate concentrations decreased to less than the EPA water quality limit of 10 mg NO_3-N L^{-1} by November of each year and remained at less than 10 mg NO_3-N L^{-1} until the next fall. Maximum labile N concentrations in the first fall were approximately 165 mg N L^{-1} for both manure+PMS treatments. In the second fall, maximum concentrations were 140 mg N L^{-1} for manure+PMS 30:1 but only 60 mg N L^{-1} for manure+PMS 20:1. In the third fall there was no such pulse and concentrations remained below 10 mg N L^{-1}.

Stehouwer et al. (2001) also reported a temporal pattern of N leaching in a Pennsylvania mine reclamation field experiment involving biosolids, with a pulse of NO_3^- lost in each fall during two years of post-reclamation monitoring. White et al. (1983) observed peak NO_3^- concentrations in the fall for sandy clay loam and clay loam soils fertilized with 135 – 144 kg ha^{-1} yr^{-1} NH_4NO_3. Based on measurements of soil water potential, they attributed the peak concentrations to enhanced mineralization induced by rewetting of the soil after summer, wherein they calculated that 23 to 28% of N losses occurred when the soil moisture differential fell from 90 mm to zero in the fall. It is likely that this phenomenon, coupled with increasing storm precipitation and the senescence of plant growth, caused the fall pulse of NO_3^- leaching observed in my experiment. Daniels et al. (2001) also observed a pulse of NO_3^- loss in the winter following application of raw biosolids or biosolids mixed with sawdust (20:1 C:N ratio) to reclaimed gravel mine soils in Virginia. They concluded, though, that enhanced NO_3^- leaching was a one time event, which contradicts results of my study showing two years of enhanced N leaching potential.

For all treatments the quantities of inorganic N losses were greatest in the first year and declined each year thereafter (Fig. 4-1, Table 4-2). However, whereas the compost and lime+fertilizer treatment losses remained relatively low and constant throughout the experiment, cumulative losses of inorganic N from the manure+PMS treatments were much larger and included the temporal pattern described above. The manure+PMS 30:1 leached more inorganic N than all other treatments in each year for a cumulative three year loss of 339 kg N ha^{-1}. While not statistically separable, the quantity of N leached from manure+PMS 20:1 appeared to be intermediate between the large losses of manure+PMS 30:1 and the small losses from the compost and the lime+fertilizer treatments. The lack of statistical power to separate the manure+PMS 20:1 treatment from the others was due to large variability among the replicate plots; in one replicate, NO_3^- concentrations were consistently two to ten times greater than in the other three replicates. If outlier data from this plot were discarded, average labile N leached from manure+PMS 20:1 would be 28.9 kg N ha^{-1}, a value much closer to losses measured from both compost and lime+fertilizer treated plots.

The manure+PMS 30:1, despite having received twice as much C as the 20:1 manure+PMS treatment, appeared to be less effective at minimizing N leaching. These results are similar to those observed in the greenhouse study, where a C:N ratio of 40:1 leached twice as much total N as the manure+PMS treatment with a 30:1 C:N ratio. Given the higher C:N ratio with the 30:1 manure+PMS, greater N immobilization, and therefore less N leaching, would have been expected (Vigil and Kissel, 1991; Parker and Sommers, 1983). It is still unclear as to why this is occurring and it appears there must be some physical or chemical inhibition of microbial activity associated with the addition of large quantities of PMS. It will likely require a great deal of additional research to elucidate the cause of this phenomenon.

While the quantity of labile N leached from manure+PMS 30:1 treatments was large, this value actually constitutes only 16% of N originally applied to the soil with this amendment (Table 4-1, 4-2). Likewise, manure+PMS 20:1 lost only 8% of N originally added. These losses are less than those observed in the greenhouse study with fresh poultry manure alone; in that study, 21% of added N was lost by leaching (see chapter II). Additionally, fresh poultry manure has been shown to lose up to 60% of added N, a significantly larger loss than was observed in the field with manure+PMS (Cabrera et al., 1993). In the previously mentioned study by Daniels et al. (2001), adding sawdust to biosolids significantly reduced N leaching losses, demonstrating that adding a high C material to fresh sewage sludge was effective at minimizing N loss. It appears that in this field study leaching losses have also been minimized by the addition of PMS to fresh manure compared to what was measured from fresh manure alone in the greenhouse experiment.

Both compost treatments lost < 1% of added N as inorganic N in leachate, demonstrating exceptional retention of N in soil and essentially no risk of N leaching to groundwater. These results are similar to those reported by Mamo et al. (1999), in which a loamy sand soil was amended with 90 Mg ha^{-1} and 270 Mg ha^{-1} of solid waste compost and both rates leached < 5% of originally added N. The extremely low losses observed from the compost treatments concur with both published literature and previous greenhouse and lab incubations studies (see chapter II and III). Compost exhibits such minimal N losses in my experiment due to N transformations that occur in the composting process, resulting in more recalcitrant, less labile N compounds (Amlinger et al., 2003). In both the greenhouse and incubation studies, compost treatments, regardless of application rate, leached less than 6% and 20%, respectively, of initially added N under leaching regimens more intense than would occur in a natural environment. In a ten year field experiment with biowaste compost, Hartl and Erhart (2005) demonstrated that N release from compost was slower than that from mineral fertilizer despite applying four times more N with compost treatments.

Lime+fertilizer treated plots, however, lost 22% of added N, even though N added with fertilizer was only 0.05 to 0.025 of the N added with the organic treatments. A study done by Zhu et al. (2003) recorded annual NO_3-N losses of 49 kg ha^{-1} with conventionally fertilized (100 kg N ha^{-1}), no-till corn production, a value twice as high as what was measured from soils reclaimed with fertilizer in this experiment. Assuming this annual rate for an agricultural system over a three year period, approximately 150 kg N ha^{-1} would be lost, comparable to losses measured from the manure+PMS 20:1 treatment over three years. Not only did a typical agricultural system lose 50% of added N, more than any treatment used in this experiment, typical no-till corn production would incur N losses of this magnitude annually while losses from any of the organic amendments tested in this experiment would be lost only in the first two years; by the third year, cumulative N losses were extremely low (< 5 kg N ha^{-1}).

Furthermore, N losses measured in this study were much smaller than have been recorded with the use of biosolids for mine reclamation, another commonly used amendment for mine reclamation. When applied at the suggested loading rate, cumulative N loss via leaching has been reported at 2327 kg N ha^{-1} over a two year period, with only 44% retention of originally added N (Stehouwer et al., 2006). This value is over six times larger than losses from manure+PMS 30:1 over three years. Given that the original concentration of N in biosolids (34.8 g N kg^{-1}) is only slightly larger than poultry manure (28.5 g N kg^{-1}) yet N leaching losses are six times higher than those measured in this experiment, the C added to fresh manure appears to play a critical role in retaining N in AML soils.

Table 4-2. Leaching losses by year and treatment. Inorganic N refers to NO_3^- + NH_4^+. Within years and columns, values that are followed by the same letter are not significantly different at $\alpha = 0.10$.

	Analyte					
Treatments	Total N	Inorg. N	Total P	Ortho-P	Total C	Organic C
	kg ha^{-1}					
Year One						
Lime+fertilizer	21.3b	22.4b	3.43a	1.07a	101b	64.7b
Compost 1	24.1b	11.3b	2.98a	1.71a	202b	134b
Compost 2	30.8b	14.5b	3.19a	1.84a	281b	190b
Manure+PMS 20:1	151b	153ab	3.99a	2.13a	345b	245ab
Manure+PMS 30:1	288a	267a	4.59a	2.90a	627a	431a
Year Two						
Lime+fertilizer	2.59b	2.28b	0.621a	0.154a	85.3c	34.0b
Compost 1	4.84b	3.20b	1.68a	0.612a	177abc	89.3ab
Compost 2	7.42b	5.41b	0.903a	0.797a	217ab	99.0a
Manure+PMS 20:1	14.0b	16.5b	1.17a	0.432a	101bc	34.0b
Manure+PMS 30:1	47.5a	66.5a	1.23a	0.388a	265a	112a
Year Three						
Lime+fertilizer	—†	0.33b	0.251a	0.152a	9.98b	7.95a
Compost 1	—	1.38b	0.990a	0.554a	56.3a	41.7a
Compost 2	—	1.68b	0.688a	0.765a	58.1a	40.0a
Manure+PMS 20:1	—	1.94b	0.442a	0.377a	26.2ab	22.6a
Manure+PMS 30:1	—	4.59a	0.432a	0.385a	59.7a	36.4a
Cumulative (3 yrs)						
Lime+fertilizer	23.9c‡	25.0b	4.21a	1.48a	197c	107c
Compost 1	28.9c	15.9b	5.27a	3.25a	435bc	265bc
Compost 2	38.2c	21.6b	4.89a	3.30a	646ab	403ab
Manure+PMS 20:1	165b	171ab	5.59a	2.95a	472bc	302bc
Manure+PMS 30:1	336a	339a	6.21a	3.72a	983a	596a

† No data available for year three

‡ Cumulative values are for the first two years

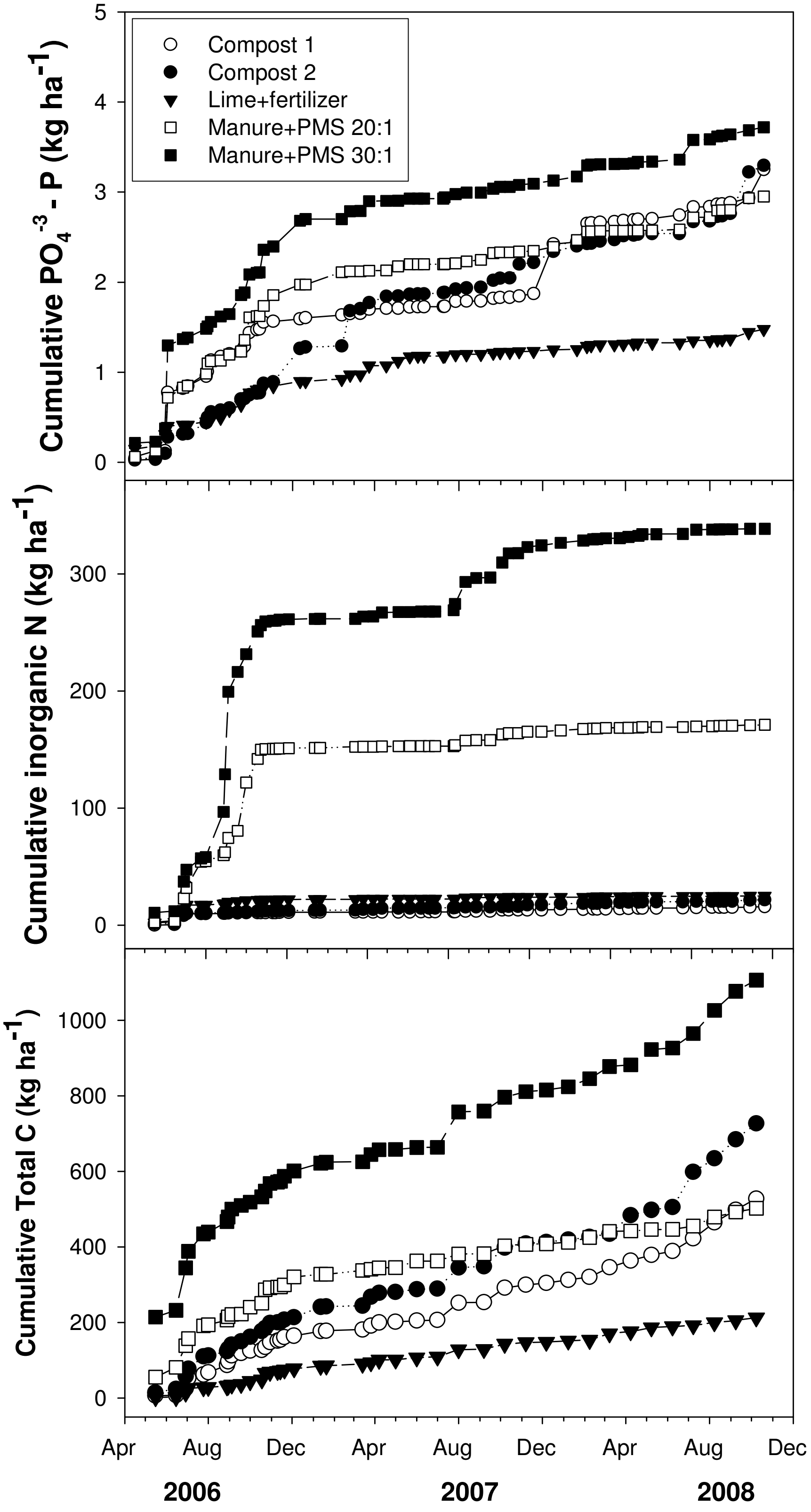

Figure 4-1. Cumulative ortho-phosphate, inorganic N (NO_3^- + NH_4^+) and total C leached over three years.

Phosphorus

Total P concentrations in leachate were initially high in the lime+fertilizer and manure+PMS 30:1 and 20:1 treatments (7.5, 6.0, and 3.0 mg P L^{-1}, respectively) during the first month after application, but remained very low throughout the rest of the experiment (< 1 mg P L^{-1}). Ortho-P followed a similar trend, with higher rates of loss in the first month, followed by a steady low rate of loss from all treatments (Fig. 4-1). On average, ortho-P losses constituted 30 to 50% of total P losses, suggesting some organic P and sediment-bound P were being transported (Stehouwer et al., 2001). There was no difference in total P or ortho-P leached among treatments in any year (Table 4-2). Therefore, regardless of application rate, all treatments were equally effective at retaining added P (> 99% retention), indicating no risk of P leaching to groundwater and implying effective sequestration in soil. These results are similar to those found in biosolids reclamation studies, where P leaching losses were also negligible compared to quantities of P added (Kostyanovskiy et al., 2008; Stehouwer et al., 2001). By contrast, studies of P loss from agricultural soils have recorded total P losses of 0.59 to 2.54 kg ha^{-1} yr^{-1} (Heathwaite et al., 2000; Jordan et al., 1997; Correll et al., 2000). Although these values are slightly lower than those measured in my experiment, agricultural losses of P are incurred on an annual basis, whereas treatments used in my study show elevated losses only in the first year after application despite P application rates five to ten times larger than the typical agronomic rate.

Carbon

Similar to P, leaching losses of C were small compared to the quantities of C added (< 2%), showing that this was not a significant loss pathway for C in these systems (Table 4-1, 4-2). There was little change in the rates of C leached throughout the course of the experiment, with manure+PMS 30:1 treatment leaching more cumulative total C than all other treatments over three years (Fig. 4-1). Leaching trends of total C largely reflect the amount of C added with each treatment. Despite a low initial addition of C, lime+fertilizer leached as much total C as compost 1 and manure+PMS 20:1, which had equal starting C values. Compost 2, although having the largest addition of C, leached less or as much total C as the other organic treatments. These results suggest the presence of a large pool of stable C in the compost that was not very susceptible to leaching losses. Labile C is greatly reduced in the process of composting, which would account for the large differences in C leached between manure+PMS and compost treatments, despite similarly large initial C inputs (Hanselman et al., 2004).

The results of this field study are similar to those observed in the lab incubation experiment, where C leaching losses represented a very small fraction of starting C and were greater for the manure+PMS 30:1 treatment in the year one experiment than all other treatments (see chapter III). In the year two incubation, there was less of a difference in C leached between manure+PMS and compost treatments, which was also observed in the field. In the incubation experiment leaching was considerably more intense and the lack of C leaching, even with extreme conditions, confirms that C is not very susceptible to loss by leaching. Although not measured in the field, significant quantities of C were undoubtedly lost as CO_2 from microbial respiration, as measured in the lab experiment. Additionally, these results point to an equally stable C pool in both manure+PMS and compost treatments by the second year after amendment application.

Organic C losses accounted for approximately 60% of total C losses over three years from organic treatments and about 50% of total C losses from the lime+fertilizer treatment. Difference in quantities of organic C lost among treatments were similar to total C, with more initial C added corresponding with more organic C lost. Again, all organic C losses were small compared to quantities initially added.

pH and Electrical Conductivity

There was a slight upward trend in leachate pH data over the three years of the experiment (Fig. 4-2). Initial pH values ranged between 5.9 and 7.4 and final values were between 6.5 and 7.8; intermediate values, although fluctuating repeatedly, fell mostly between the lower and upper limits of the initial and final pH ranges (5.9 to 7.8). Given the initial soil pH of 5.1 and the near surface application of all amendments, this upward trend in leachate pH suggests that pH has been effectively raised with depth. Moreover, these treatments appear to be very effective at raising pH in the acidic mine soil and acidic water is not leaching from these soils, at least in the upper 30 cm.

Electrical conductivity (EC) trends were similar to labile N leaching concentrations: values were highest in October of 2006 and 2007 for the manure+PMS treatments, corresponding with pulses of N leached from these plots (Fig. 4-2). Compost 2 also had higher EC levels in the first month after amendment application and much lower EC thereafter. The decrease in EC over time indicates greater losses of nutrients early on in the experiment, with losses decreasing over time.

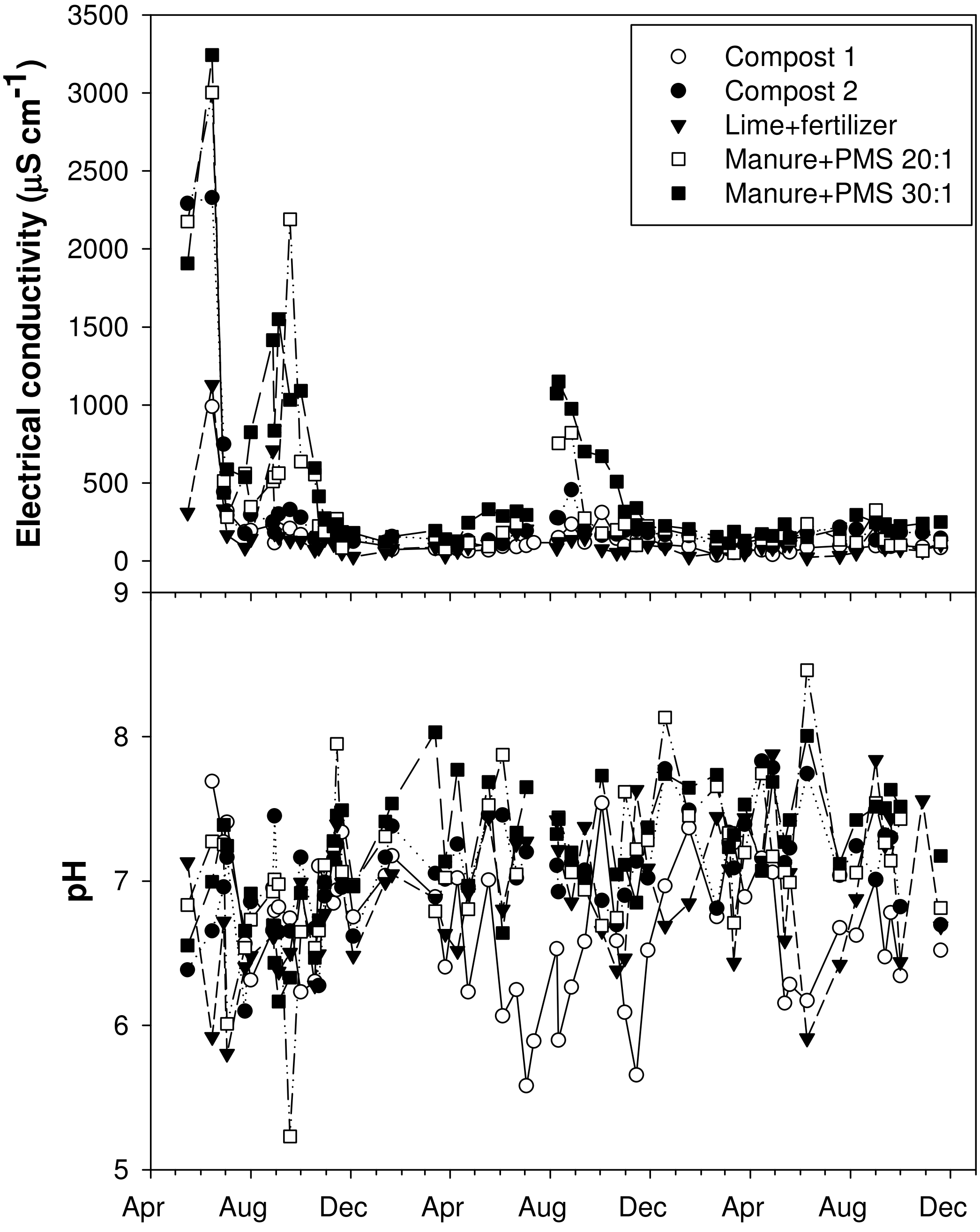

Figure 4-2. Electrical conductivity and pH measured in leachates by treatment over three years.

Mine soil chemistry

Soil pH

Soil pH responded rapidly to all amendment applications; within two months pH had increased from 5.1 to 7.0 for all organic amendments and from 5.1 to 6.4 for lime+fertilizer (Fig. 4-3). Thereafter, soil pH increased slightly to a final pH of 7.4 in organically amended plots and 6.8 in lime+fertilizer plots, well within the ideal range for plants. Given the substantial calcium carbonate equivalence (CCE) of the organic amendments, significantly more alkalinity was applied with the organic amendments than the lime+fertilizer treatment (Table 4-1). The slight increase in pH over time also suggests the liming effect was not a temporary artifact of amendment addition.

Although the starting soil pH was not overly acidic so as to completely inhibit vegetative growth, it likely allowed for some metal mobility and was not ideal for grass production, especially at an intensive level as would be necessary for switchgrass cultivation as a biofuel. All of these treatments were very good for raising pH and ameliorating soil phytotoxicity. These results are similar to a study by Eghball and Gilley (1999), in which manure, compost and inorganic fertilizer were applied to a silty clay loam field soil. They measured an increase in pH from 5 to 7.3 with all treatments, confirming that these materials are adequate for raising soil pH. The previously conducted greenhouse experiment also showed similar increases in mine soil pH with these amendments (see chapter II).

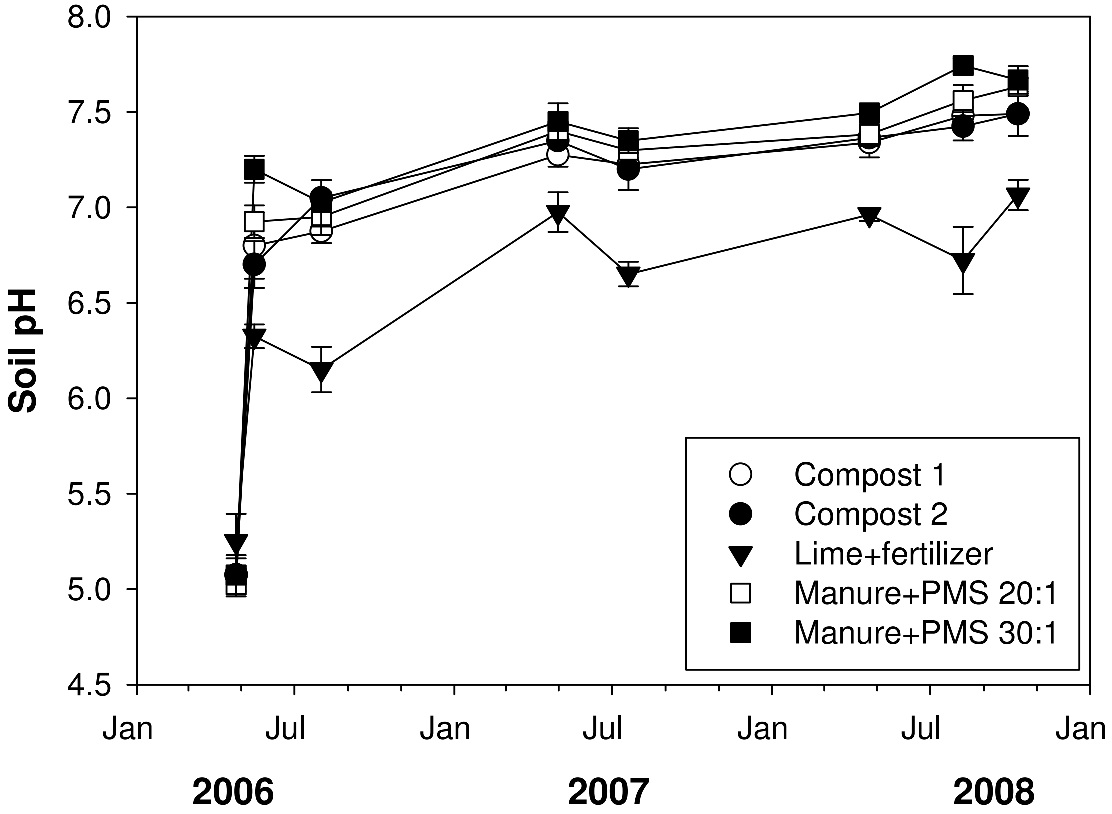

Figure 4-3. Soil pH by treatment over three years. Error bars represent one standard error of the mean.

<u>Soil C</u>

There was a distinct increase in soil C observed over time (Fig. 4-4). Initial soil C concentrations were approximately 18 g kg^{-1} prior to adding treatments; soil C concentrations rapidly increased after amendment addition as expected based on the large quantities of C associated with the amendments. There was also some seasonal variation with more total C measured in spring compared to summer. All organic amendments showed a clear trend of increasing soil C concentrations with time, whereas the lime+fertilizer treatment exhibited a very small increase. This increase demonstrates the ability of these treatments to store C in the soil better than a conventional inorganic fertilizer treatment.

Two years after amendment incorporation, the quantity of C sequestered in soil was greatest for compost 2 (55.7 Mg ha^{-1}); all other organic treatments also sequestered C, although slightly less (approximately 40 Mg ha^{-1}) (Table 4-3). The lime+fertilizer treatment had the lowest quantity of soil C after two years, implying less C sequestration compared to organic amendments (31.2 Mg ha^{-1}). The lack of statistical power to separate the manure+PMS and compost 1 treatment from the lime+fertilizer, however, was due to large variability among replicate plots. In a study by Garten and Wullschleger (1999) examining soil organic carbon (SOC) accumulation under different types of plant cover, including switchgrass, corn, and tall fescue five years after planting, a sample size of n > 100 was deemed necessary to statistically separate SOC measurements. It appears that, in order to obtain a better grasp on actual C sequestration potential with these organic treatments, a more intense soil sampling method should be considered.

At a depth of 10 cm there was no difference among treatments in the quantity of C sequestered: soil C was approximately 15 Mg ha^{-1}. The C increase in all treatments was a result not only of amendment addition, but also the incorporation of biomass and accumulation of root mass over time, which would be expected to increase further given the immaturity of the switchgrass stand (2 yr). Ma et al. (2000) demonstrated a positive relationship between root input and soil C accumulation and found that C mineralization, microbial biomass C and C turnover were all measurably higher in the second year after planting than immediately following establishment in a sandy loam soil. Although these data are clearly very preliminary, results of the incubation experiment also showed larger stable C pools with all organic amendments compared to the lime+fertilizer treatment, further substantiating the idea of greater C sequestration potential with the use of organic amendments. Again, additional monitoring is necessary to conclusively determine C sequestration potential, but preliminary data look promising.

Table 4-3. Quantity of soil total N and C prior to amendment application (initial) and after 3 years (final). Within columns values followed by the same letter are not significantly different at $\alpha = 0.10$.

Treatment	Soil N		Soil C	
	Initial	Final	Initial	Final
	——— $kg\ ha^{-1}$ ———		——— $Mg\ ha^{-1}$ ———	
Lime+fertilizer	790a	1150c	22.5a	31.2b
Compost 1	790a	2880b	18.9ab	39.6b
Compost 2	767a	4780a	17.9ab	55.7a
Manure+PMS 20:1	835a	1940bc	16.6b	40.5b
Manure+PMS 30:1	812a	2060bc	17.5b	41.3b

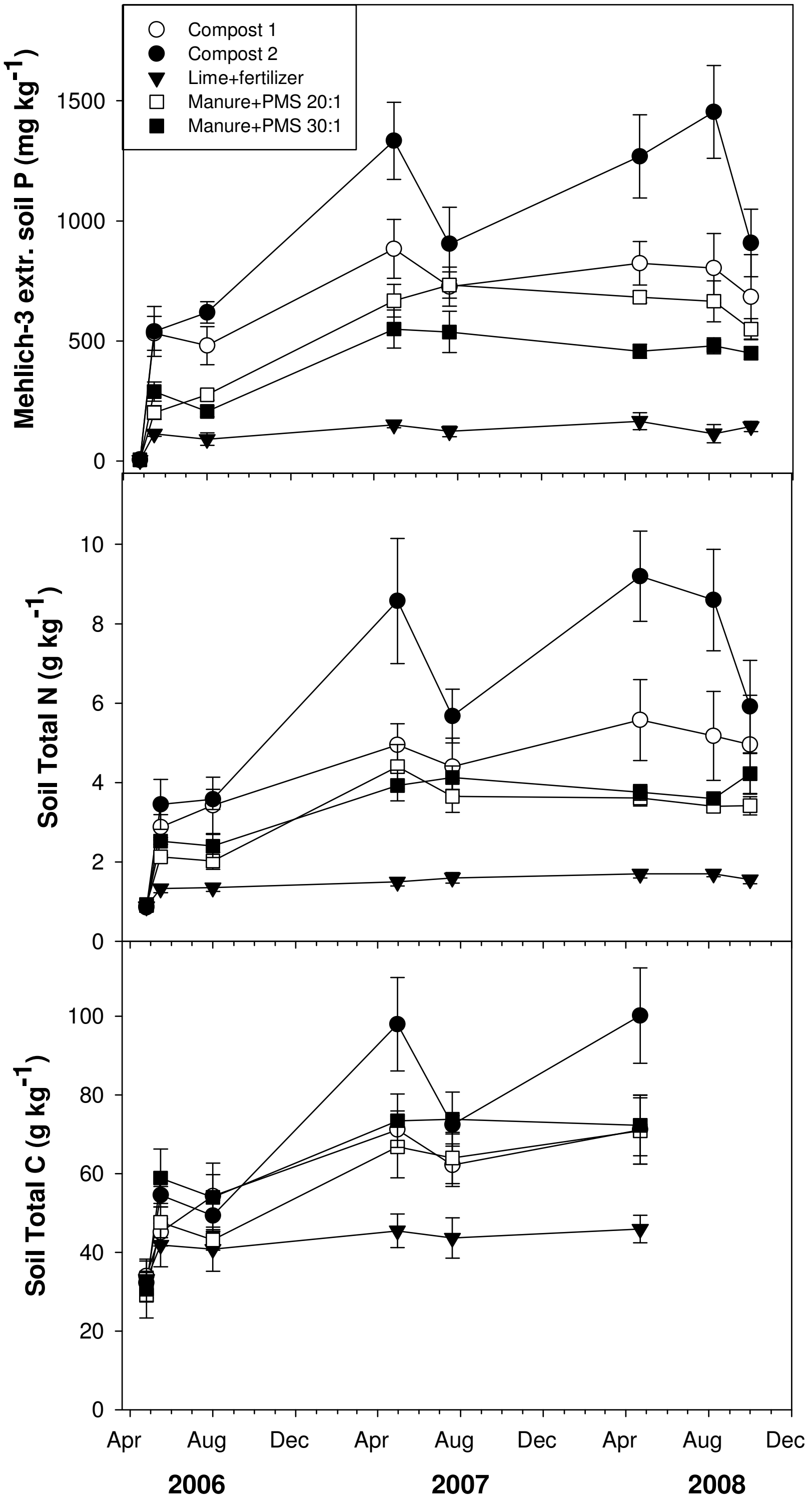

Figure 4-4. Mehlich-3 extractable soil P, soil N and soil C by treatment over three years. Bars represent standard errors of the means. No data was available May through November 2008 for soil C.

Soil P

Concentrations of Mehlich-3 extractable soil P also increased with time for all treatments, although changes observed with the lime+fertilizer treatment were minimal (Fig. 4-4). Organic amendments showed a two- to three-fold increase over three years, with compost 2 retaining more plant available P than all other organic treatments (505 kg ha^{-1}). At a depth of 10 cm, there was no difference in soil P among treatments (approximately 200 kg ha^{-1}).

Although these levels of soil P are quite high, they do not appear to be at risk for leaching, as very little P was leached from any treatments with accumulation of plant available P in the soil. It is likely that the addition of $CaCO_3$ in the PMS resulted in the precipitation of Ca phosphates as the soil pH increased following amendment addition, which further reduced the amount of soluble P available for leaching (Havlin et al., 1999). Or, P was possibly bound to organic matter or other soil constituents (such as Fe or Al) and therefore was not subject to removal by leaching (Kostyanovskiy et al., 2008). There is also minimal loss of P by erosion, as the switchgrass is a perennial crop that, even when harvested, will not leave the soil uncovered. Sanderson et al. (2001) measured reductions of up to 95% of total reactive P in surface runoff when 100 kg N ha^{-1} was applied as dairy manure effluent to a switchgrass buffer strip, demonstrating that even with raw manure application, P runoff was reduced by having a perennial crop.

Soil N

Total soil N concentrations increased immediately following amendment application and continued to increase for organic amendments, but remained constant for the lime+fertilizer treatment (Fig. 4-4). There were also seasonal differences in soil N between the spring and summer sampling dates. Three years after amendment application, more soil N was measured in the compost treatments while lime+fertilizer treated soils sequestered less N (Table 3). Manure+PMS treatments, although not statistically separable from the lime+fertilizer treatment, appears to have also accumulated more soil N over time (Fig. 4). Increased soil N sequestration corresponds well with minimal N leaching of 1% (compost treatments) and 8 and 16% (manure+PMS 20:1 and 30:1, respectively) of originally added N. Considering the large quantities of N added with these organic treatments, 10 to 20x more than the lime+fertilizer treatment which is a typical agronomic rate for warm season grasses in PA, the lack of leachate loss implies significant N sequestration in soil, which this data supports (Table 1).

While these data are also very short term, trends are undoubtedly telling of greater sequestration potential, and more N available for plants, with organic amendments compared to inorganic in the long term. This N pool is especially important to sustain high switchgrass yields while minimizing the need for frequent N inputs, as is required in typical agronomic systems. With the lime+fertilizer treatment, it does not appear that a large soil N pool will be available for switchgrass. The lab incubation also showed larger stable N pools with organic amendments, confirming implied N sequestration trends observed in this field study (see chapter III).

Soil K

Immediately following amendment application, Mehlich-3 extractable soil K concentrations increased substantially (150- to 250-fold) in organic amendments but only

50-fold for the lime+fertilizer treatment (Fig. 4-5). Levels of plant available K greatly exceeded the optimum range of 100 to 200 mg kg^{-1} with the organic treatments, but soil K rapidly declined by fall 2006 and continued to decrease in all organically amended soils to optimum levels. Lime+fertilizer also showed a decline four months after amendment addition but showed no further decreases thereafter. After three years, soil K concentrations were greater than initial concentrations prior to amendment addition, but it appears that a large quantity of added K was lost in the first year given the large quantities of K added (Table 1). Despite this leaching loss of K, however, all treatments had optimal levels of plant available soil K after three years, suggesting that all of these treatments were effective at adding K and improving soil K from below optimum levels of 4 mg kg^{-1} measured before amendment addition.

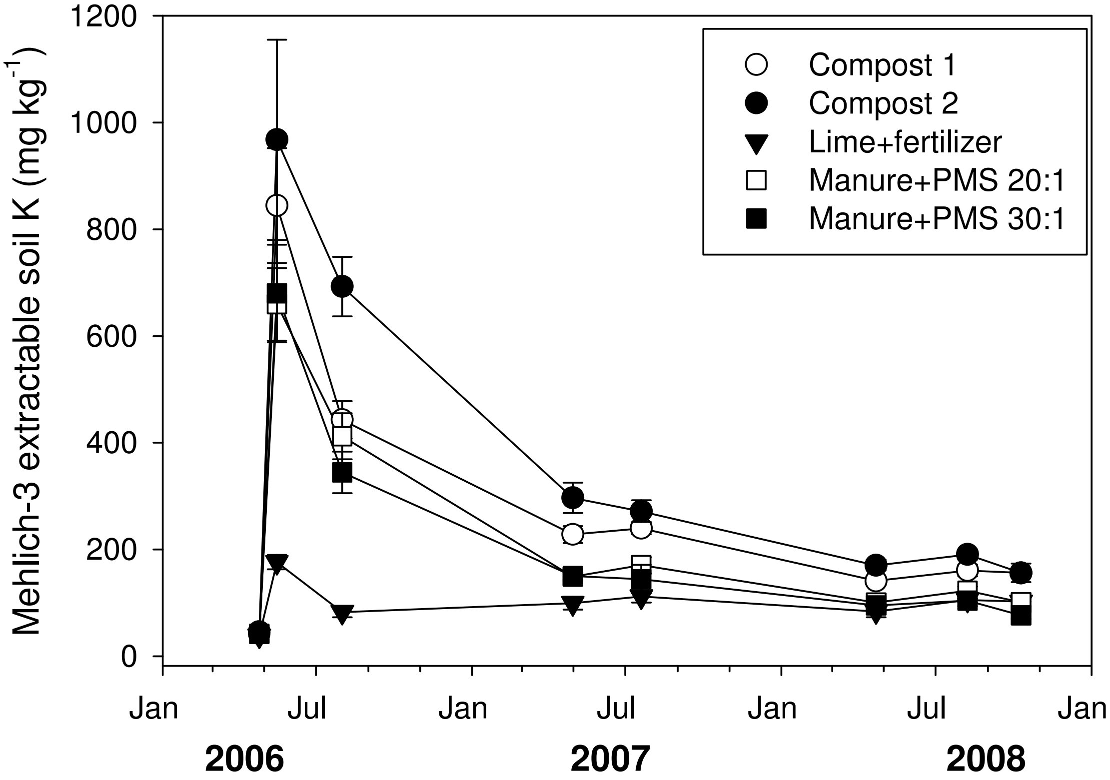

Figure 4-5. Mehlich-3 extractable soil K by treatment over three years.

Switchgrass growth

Biomass

In 2006, good vegetative cover was established with all treatments. There was no effect of treatment on biomass, demonstrating that all treatments were equally effective at ameliorating phytotoxic conditions and establishing vegetative cover (Fig. 4-6). Vegetative cover was quickly established in the first two months but the ryegrass, intended as a nurse crop for the switchgrass, outcompeted the latter and inhibited its establishment until the second year. Switchgrass seed dormancy often inhibits rapid establishment of switchgrass, especially in competitive situations (Sanderson et al., 1996). In 2007, biomass was much less than in 2006, reflecting the transition in plant populations from ryegrass to switchgrass. Despite the overall low yields (< 160 g m^{-2}), there were small differences among treatments.

In 2008, biomass was even greater than in the previous two years, with compost and manure+PMS treatments exhibiting yields over 3x larger than lime+fertilizer. Yields for the organic treatments in 2008 are similar to those reported by McLaughlin et al. (2004) three years after switchgrass (var. Alamo) establishment in a silty clay soil fertilized with swine effluent, which applied 371, 61, and 629 kg ha^{-1} of N, P and K, respectively; their rates were much smaller for N and P, but K was greater, than those used in this experiment. The results of my field study reflect a two-year stand of switchgrass and demonstrate the ability of minesoils to support high yields with poultry manure and a high C material compared to a simple application of lime and fertilizer. Given that three to five years is required to establish a mature stand of switchgrass, these data, although preliminary, show great potential for amended soils to support high yields

(McLaughlin and Kszos, 2005). Additionally, the larger soil N and C pools observed with the organic treatments over time imply greater nutrient retention that could support large stands of switchgrass beyond the first few years (Table 4-4). Further monitoring of biomass production, coupled with research on the effects of switchgrass harvesting, is needed to determine the sustainability of switchgrass production in these soils.

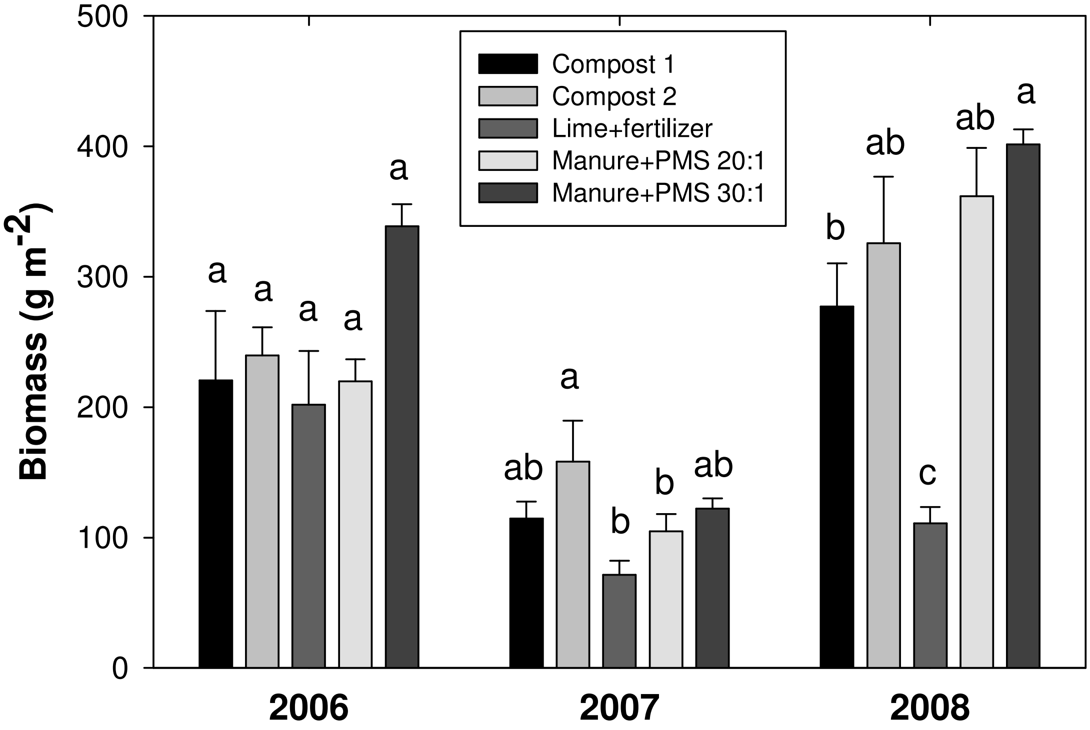

Figure 4-6. Biomass yield by year and treatment. Grass population in 2006 was dominated by annual ryegrass; 2007 was a mix of annual ryegrass and switchgrass. 2008 biomass was dominated by switchgrass. Same letters indicate no significance difference at α=0.05.

Tissue analysis

There were no differences among treatments for tissue macronutrients (N, P, K and Ca) by year (Table 4). Tissue concentrations were initially excessive for all macronutrients except Ca, but have since decreased to more typical agronomic levels (Reid et al., 1992). Decreasing concentrations of N and K over three years were likely due to increasing biomass, which would effectively dilute nutrient concentrations, coupled with luxury consumption by plants, especially in the case of K. Micronutrient concentrations did vary by treatment. In 2006, concentrations of Al, Cu, Fe, Na, and Zn were significantly greater in manure+PMS treatments than in compost or lime+fertilizer. B concentrations during this year were higher in all organic amendments compared to the control. In 2007 and 2008, compost treatments had greater concentrations of B, Na and Zn. Fe concentrations remained greater in manure+PMS treatments in 2007 and 2008.

In comparison to McLaughlin et al. (2004), tissue concentrations measured in this experiment were very similar, except for Mn and Zn, which were much greater here. Zn was the only micronutrient that was excessively high, especially in the first year, but concentrations have dropped considerably closer to optimal levels by the third year after planting. In no year, however, did macro- or micro- nutrient levels seem unsuitable for plant growth, as levels were largely within normal ranges and did not have any noticeable impact on switchgrass yields.

Table 4-4. Tissue analysis by year and treatment. Within years and columns values followed by the same letter are not significantly different at α = 0.10.

	Macronutrients				Micronutrients						
Treatments	N	P	K	Ca	Al	B	Cu	Fe	Mn	Na	Zn
	g kg^{-1}				mg kg^{-1}						
2006											
Lime+fertilizer	20.9a	3.58a	32.4a	3.56a	16.0bc	4.75b	5.75c	43.5b	—†	168c	190c
Compost 1	20.5a	3.80a	29.6a	3.40a	12.3c	7.75a	5.00c	43.8b	—	111c	176b
Compost 2	14.5a	3.78a	27.3a	2.45a	10.0c	8.75a	5.00c	45.8b	—	141c	275b
Manure+PMS 20:1	20.2a	3.75a	28.8a	3.33a	45.3ab	8.75a	9.75b	71.8a	—	643b	277b
Manure+PMS 30:1	20.4a	3.45a	26.2a	3.43a	65.3a	8.00a	13.0a	78.3a	—	967a	222a
2007											
Lime+fertilizer	18.0a	2.83a	16.6a	3.62a	—†	3.30c	9.93a	54.6b	197a	64.4b	48.6a
Compost 1	19.1a	2.75a	16.6a	3.99a	—	7.18a	7.80bc	64.8b	129bc	82.7ab	45.2a
Compost 2	16.8a	3.95a	17.9a	3.74a	—	5.76b	7.78bc	61.7b	90.5c	106a	45.0a
Manure+PMS 20:1	19.5a	3.18a	14.0a	6.50a	—	5.30b	9.11ab	98.9a	192ab	62.7b	34.1b
Manure+PMS 30:1	18.3a	2.35a	12.9a	4.88a	—	5.21b	6.66c	98.8a	76.2c	65.2b	23.6c
2008											
Lime+fertilizer	10.6a	2.88a	8.30a	4.10a	—†	3.25c	3.25a	34.0b	201a	15.5b	37.8a
Compost 1	10.4a	2.33a	8.78a	4.35a	—	4.25a	5.25bc	38.0b	82.3bc	20.2ab	39.3a
Compost 2	12.2a	3.58a	10.2a	4.50a	—	2.75b	6.00bc	36.0b	139c	22.3a	48.0a
Manure+PMS 20:1	9.25a	2.48a	8.70a	4.20a	—	2.25b	5.25ab	30.5a	131ab	18.0b	33.8b
Manure+PMS 30:1	8.95a	2.13a	7.90a	3.90a	—	2.00b	5.00c	31.8a	105c	18.8b	25.8c
Optimal levels‡	13.2	2.20	16.3	3.9	—§	—§	6.00	58.0	123	—§	22

† Not analyzed

‡ Data from Reid et al. (1992)

§ No data available

Conclusions

Among all treatments in this study, compost treatments were most effective at retaining N, providing an environmentally sound reclamation method, especially in the sensitive Chesapeake Bay watershed. Leaching losses of N from these treatments constituted less than 1% of N added with the compost treatments despite extremely high N application rates (2117 and 4234 kg ha^{-1}). Fresh layer manure mixed with paper mill sludge in a 20:1 C:N ratio leached more N than compost, but was still better at retaining N than the 30:1 C:N manure+PMS treatment, a result observed in the earlier greenhouse study.

Although leaching losses were greater from manure+PMS treatments than those from compost treatments, these methods pose a significantly lower environmental risk than reclamation with biosolids, which has been shown to leach up to 7x more N than the manure+PMS 30:1 treatment when applied at the recommended loading rate for mine reclamation (Stehouwer et al., 2006). The large reduction in leaching losses compared to biosolids application also corroborates results of the greenhouse study that demonstrated adding a high C material increased N retention. Furthermore, these treatments incurred N losses only in the two years following amendment application, demonstrating a very short term risk of N leaching to groundwater. Agricultural N applications, however, can lose up to 50 kg N ha^{-1} yr^{-1}, or half of N applied as inorganic fertilizer (Zhu et al., 2003). These losses occur on an annual basis and therefore pose a greater long term risk of environmental N pollution than any of the treatments used in my study.

All treatments also produced sufficiently high switchgrass yields to revegetate the mine site. Compared to traditional reclamation methods of adding lime and fertilizer, however, composted manure or manure+PMS produced superior vegetative growth three years after amendment application. Nutrient sequestration data, although very short term, also suggested increased retention of nutrients with organic treatments over time compared to the lime+fertilizer treatment. In sum, both compost and manure+PMS treatments were superior alternative reclamation methods that effectively sequestered nutrients and improved switchgrass yields. It also appears that N application rates in the manure+PMS treatments could be reduced to further reduce N leaching potential while maintaining high switchgrass yields. These results have implications for the long-term enhancement of soil quality and sustainable biomass production.

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Chapter V

Conclusions

All treatments examined in this research, including lime plus fertilizer, fresh poultry manure, composted poultry manure, and fresh manure mixed with paper mill sludge, were equally effective at ameliorating soil phytotoxicity of mine soils and producing vegetative cover. Fresh manure alone leached significantly more nutrients than any treatment, whereas leaching losses were considerably smaller for treatments containing both fresh manure and paper mill sludge. These results confirm that C:N ratio adjustment of poultry manure with paper mill sludge is an effective way to minimize nutrient losses. In all three experiments, the application of composted poultry manure to mine soil proved to be an extremely effective method for minimizing nutrient loss and sequestering more nutrients in the soil, confirming that this method of mine reclamation poses minimal environmental risks.

Organic amendments generated substantially larger stable N and C pools than the lime plus fertilizer treatment, implying better restoration of soil productivity and sustainability of nutrient cycling in these nutrient poor mine soils compared to the lime plus fertilizer treatment. Under intense leaching conditions, manure and paper mill sludge treatments were just as effective as compost treatments at minimizing N leaching and actually generated larger microbial pools. The enhanced microbial activity with manure and paper mill sludge treatments could translate to enhanced, long term soil nutrient cycling.

Switchgrass yields were significantly higher for all organic treatments compared to the lime plus fertilizer treatment three years after amendment application, demonstrating that a lime plus fertilizer treatment would be insufficient to produce high switchgrass yields necessary for economical biofuel production on mine soils. Furthermore, it appears that manure and paper mill sludge application rates could be further reduced to more effectively minimize nutrient losses while maintaining high switchgrass yields.

The economics of mine reclamation necessitate an inexpensive method for widespread adaptation and successful implementation. On-site application of manure and paper mill sludge would fulfill this stipulation more so than compost, which incurs additional expenses during the production and transport of material. Finally, the production of switchgrass or participation in a C or N sequestration program could help pay for the reclamation costs incurred with these methods.

Appendix A

Supplemental Data for Chapter II

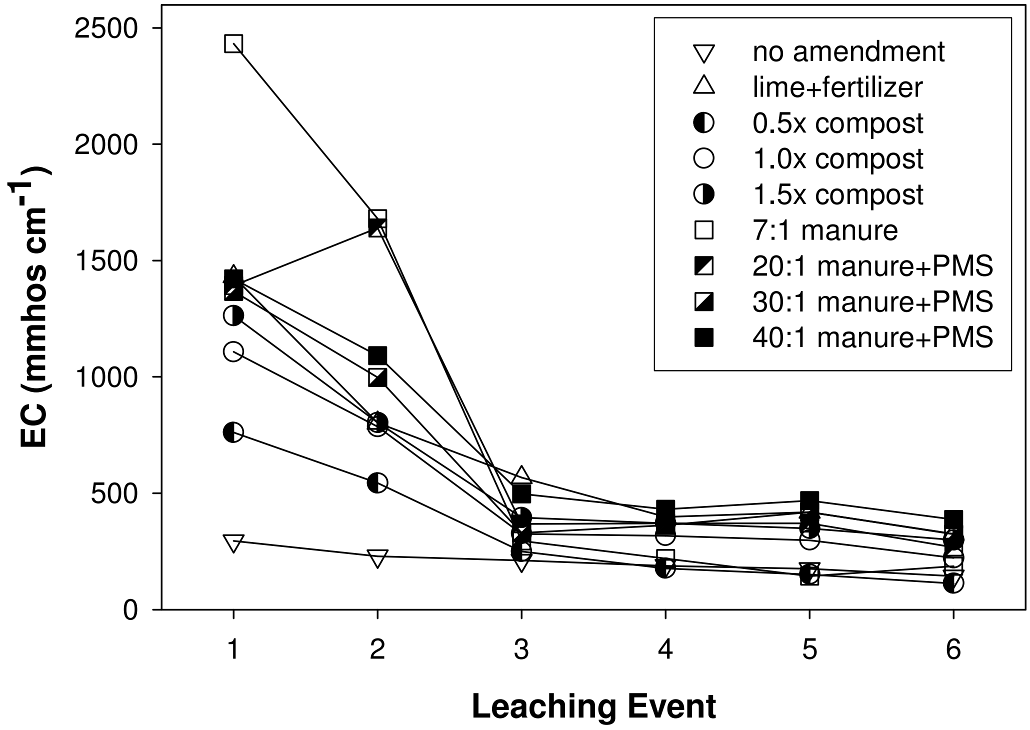


Figure 1. Electrical conductivity measurements of leachate at each of six leaching events by treatment.

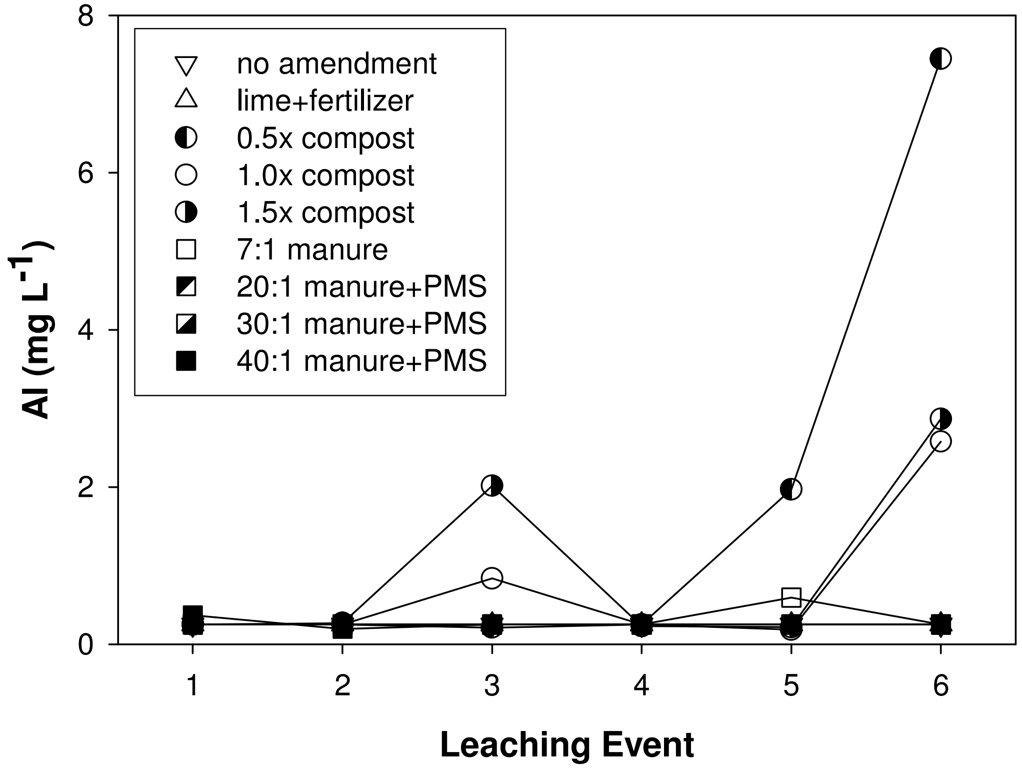


Figure 2. Aluminum concentrations in leachate at each of six leaching events by treatment.

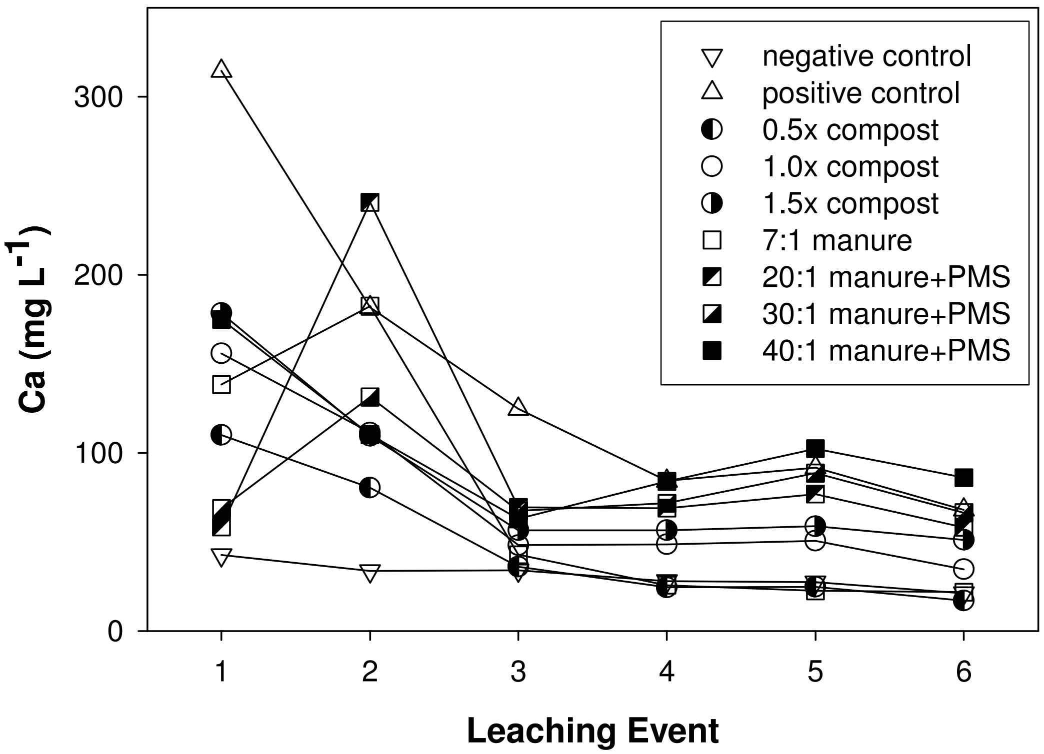


Figure 3. Calcium concentrations in leachate at each of six leaching events by treatment.

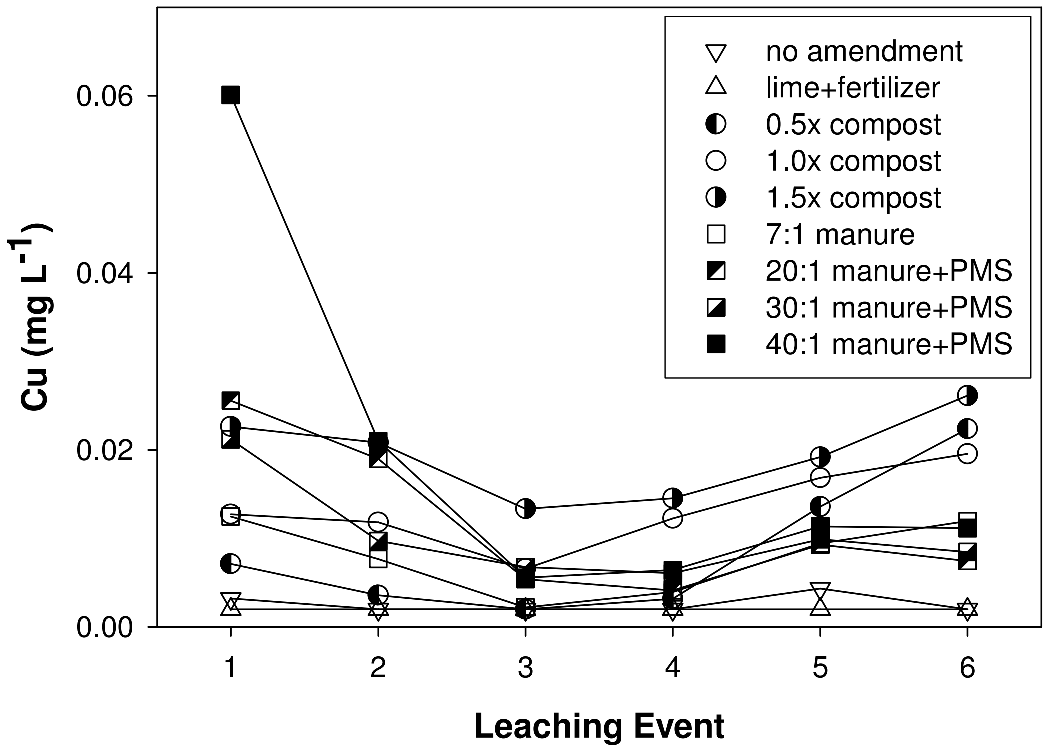


Figure 4. Copper concentrations in leachate at each of six leaching events by treatment.

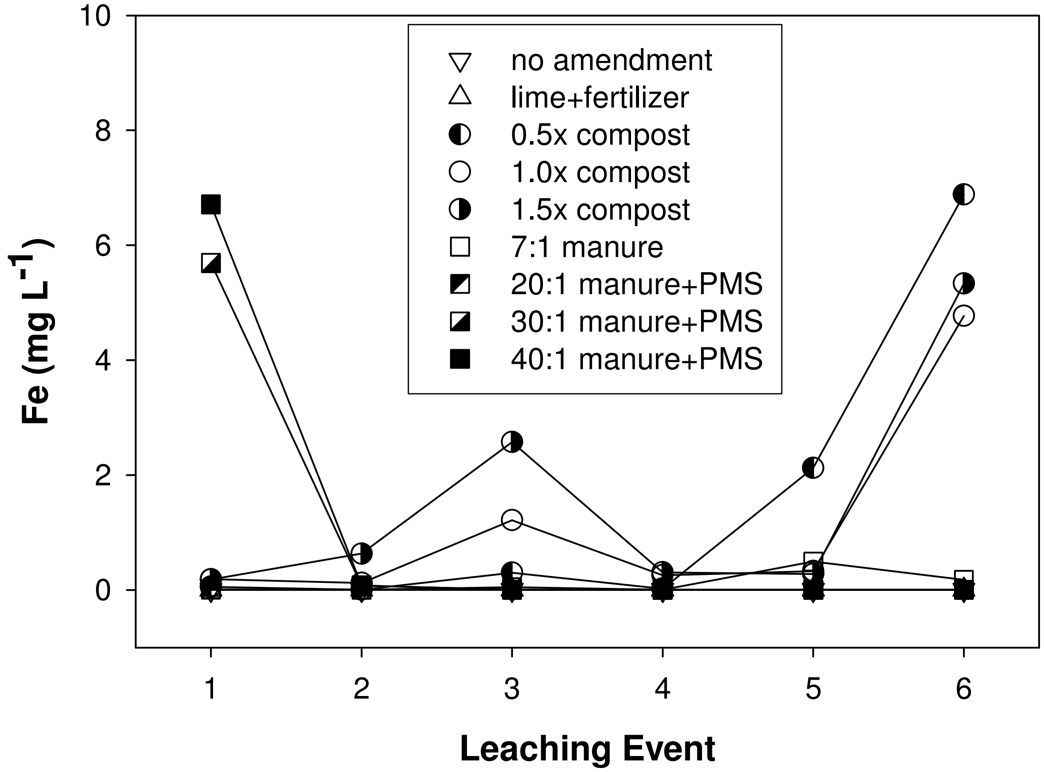


Figure 5. Iron concentrations in leachate at each of six leaching events by treatment.

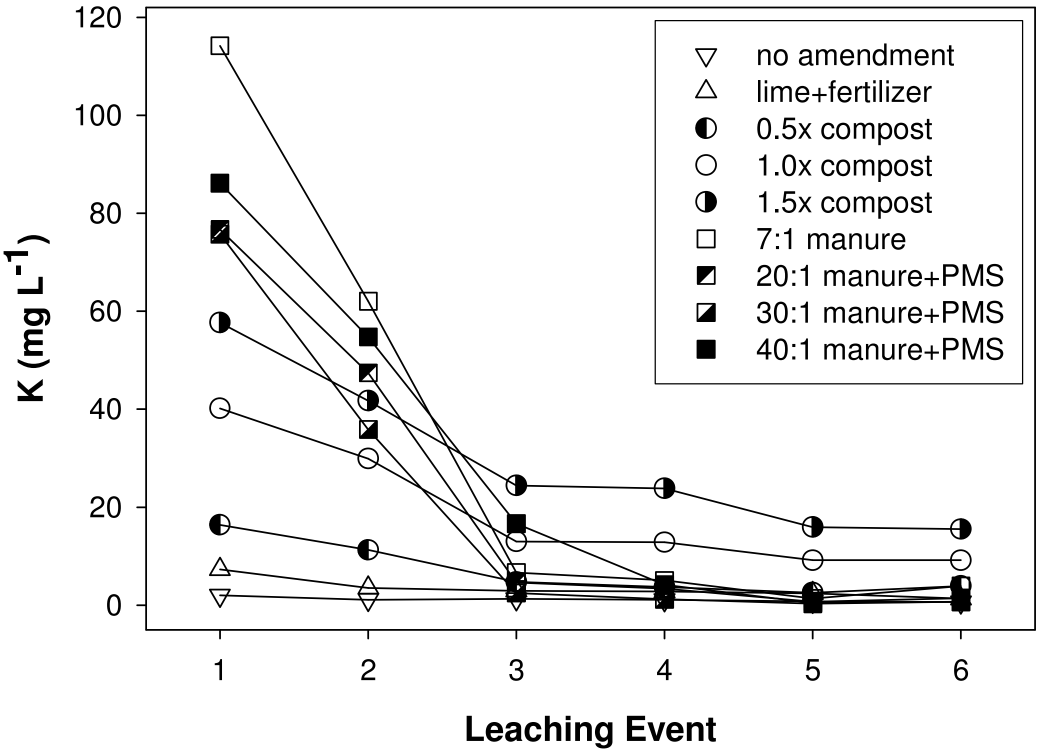


Figure 6. Potassium concentrations in leachate at each of six leaching events by treatment.

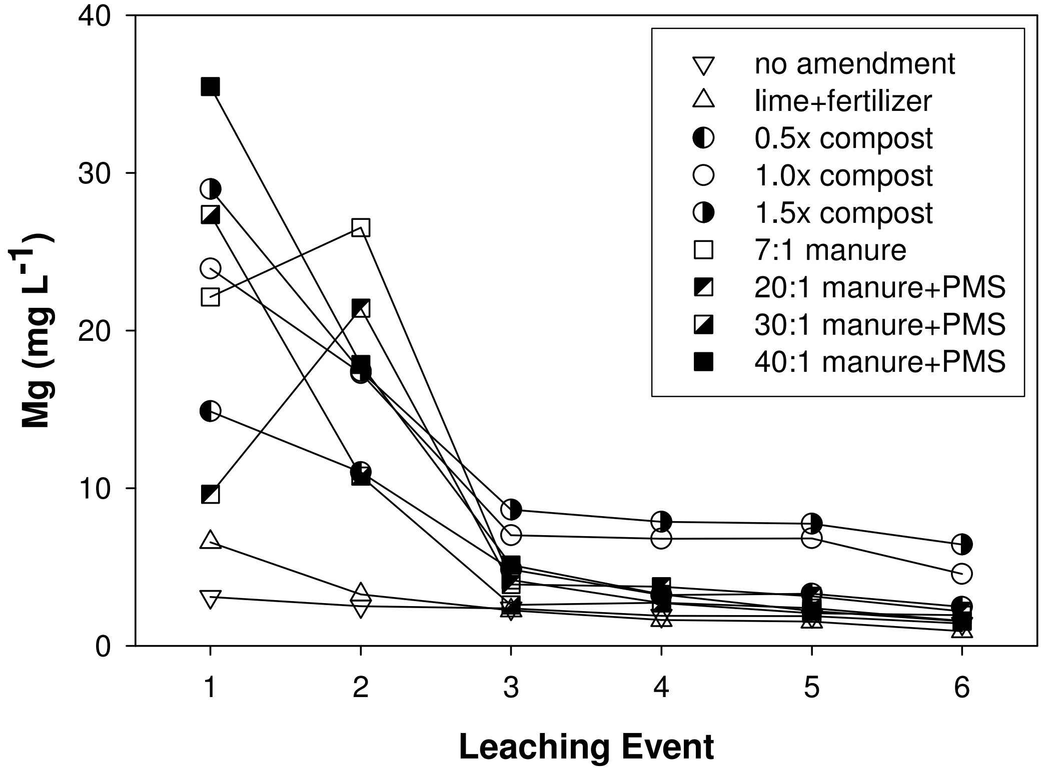


Figure 3. Magnesium concentrations in leachate at each of six leaching events by treatment.

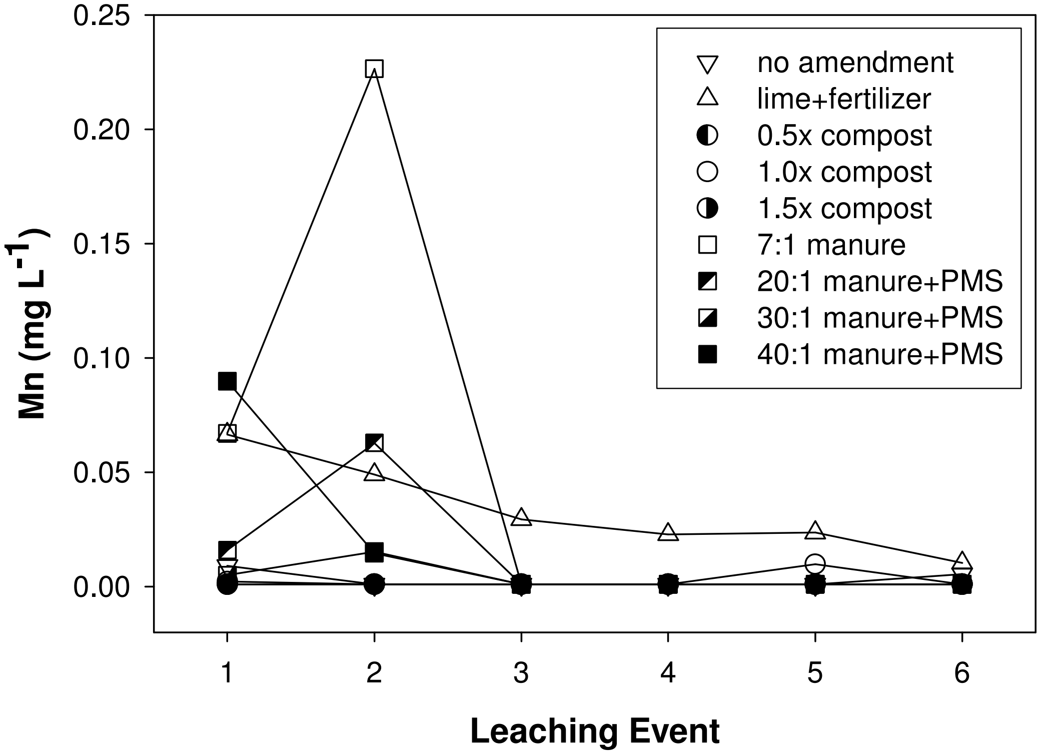


Figure 8. Manganese concentrations in leachate at each of six leaching events by treatment.

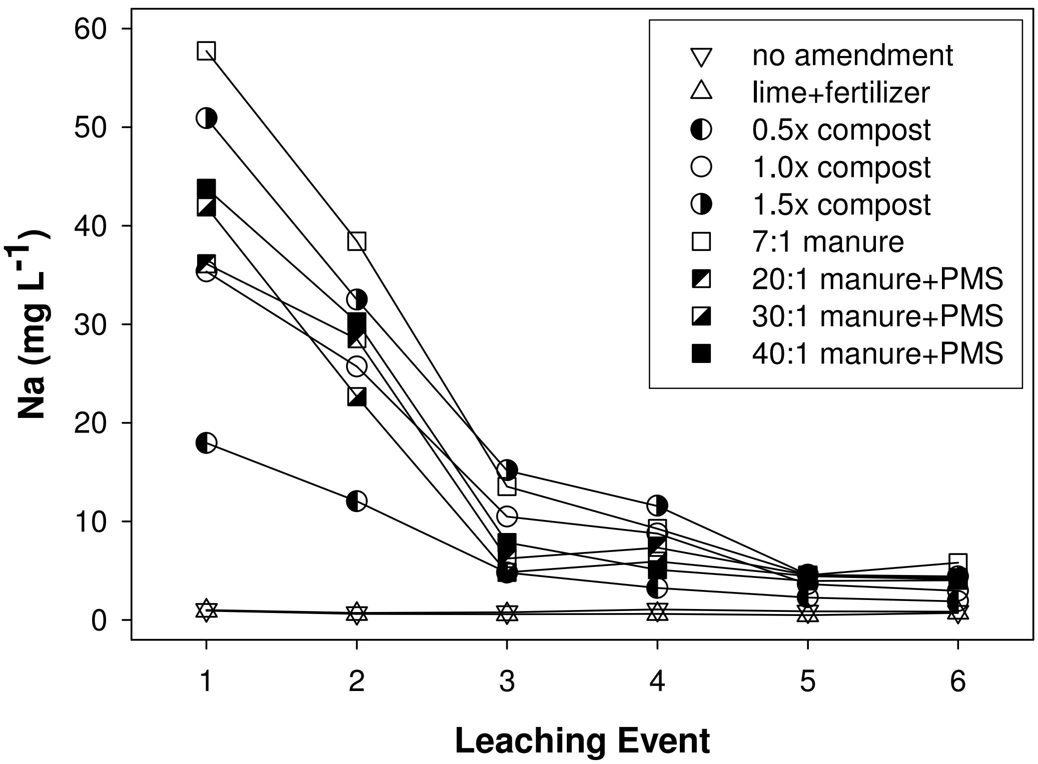


Figure 9. Sodium concentrations in leachate at each of six leaching events by treatment.

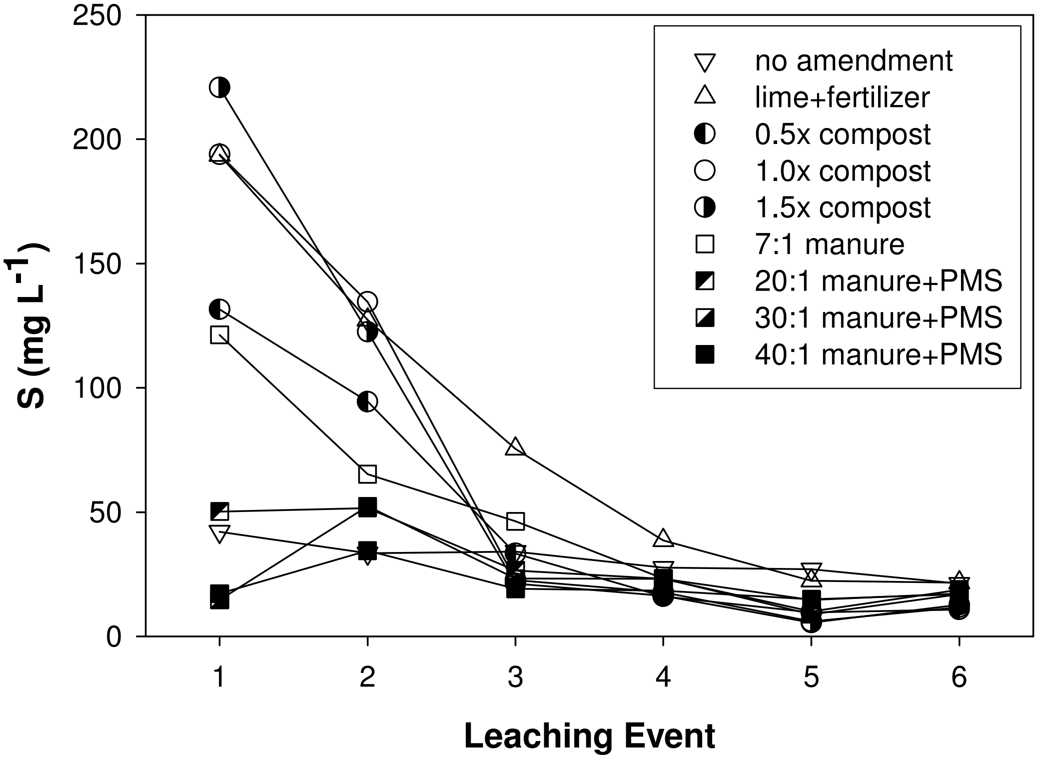


Figure 10. Sulfur concentrations in leachate at each of six leaching events by treatment.

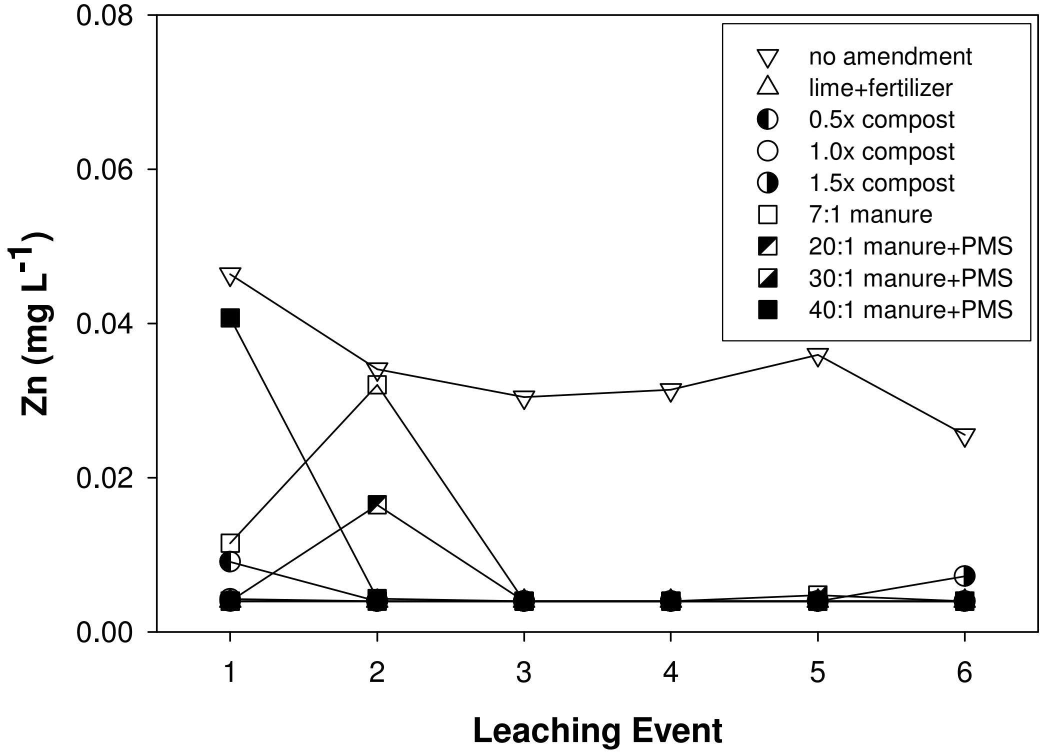


Figure 11. Zinc concentrations in leachate at each of six leaching events by treatment.

Appendix B

Supplemental Data for Chapter III

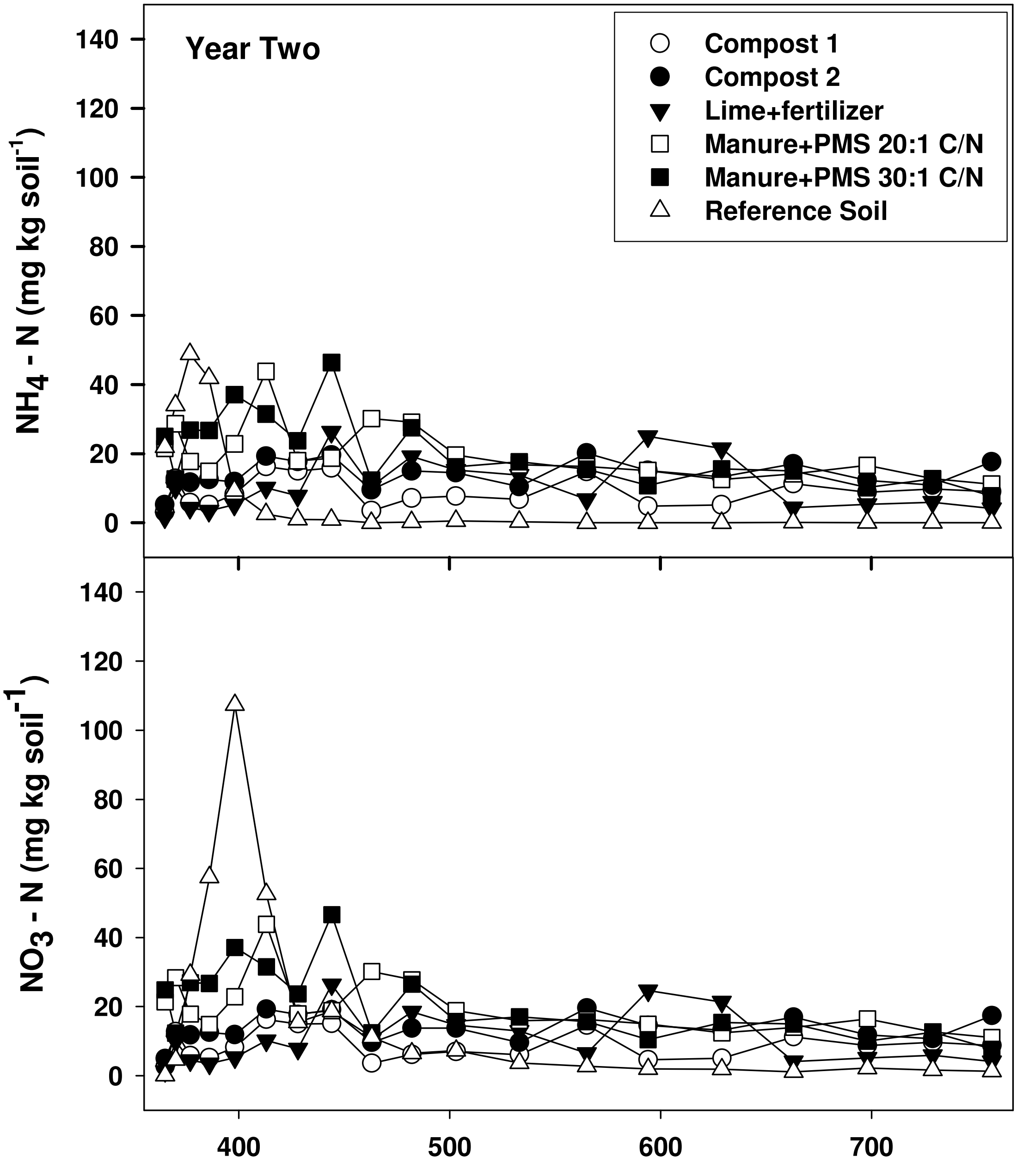
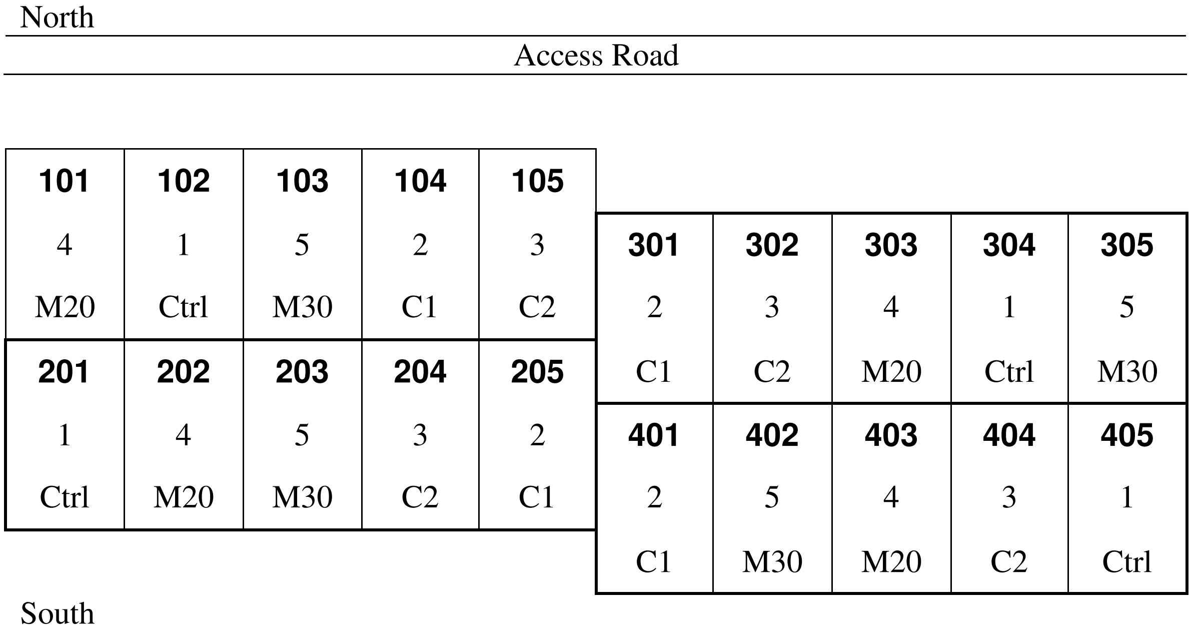


Figure 12. Ammonium and nitrate leached during the year two incubation (365 - 757 days).

Appendix C

Supplemental Data for Chapter IV



101 4 M20	**102** 1 Ctrl	**103** 5 M30	**104** 2 C1	**105** 3 C2
201 1 Ctrl	**202** 4 M20	**203** 5 M30	**204** 3 C2	**205** 2 C1

301 2 C1	**302** 3 C2	**303** 4 M20	**304** 1 Ctrl	**305** 5 M30
401 2 C1	**402** 5 M30	**403** 4 M20	**404** 3 C2	**405** 1 Ctrl

Treatments

1	Control (Lime + fertilizer)
2	Compost 1 (30 T/A)
3	Compost 2 (60 T/A)
4	Manure/PMS (20:1)
5	Manure/PMS (30:1)

Figure 13. Diagram of field plot design and associated treatments.

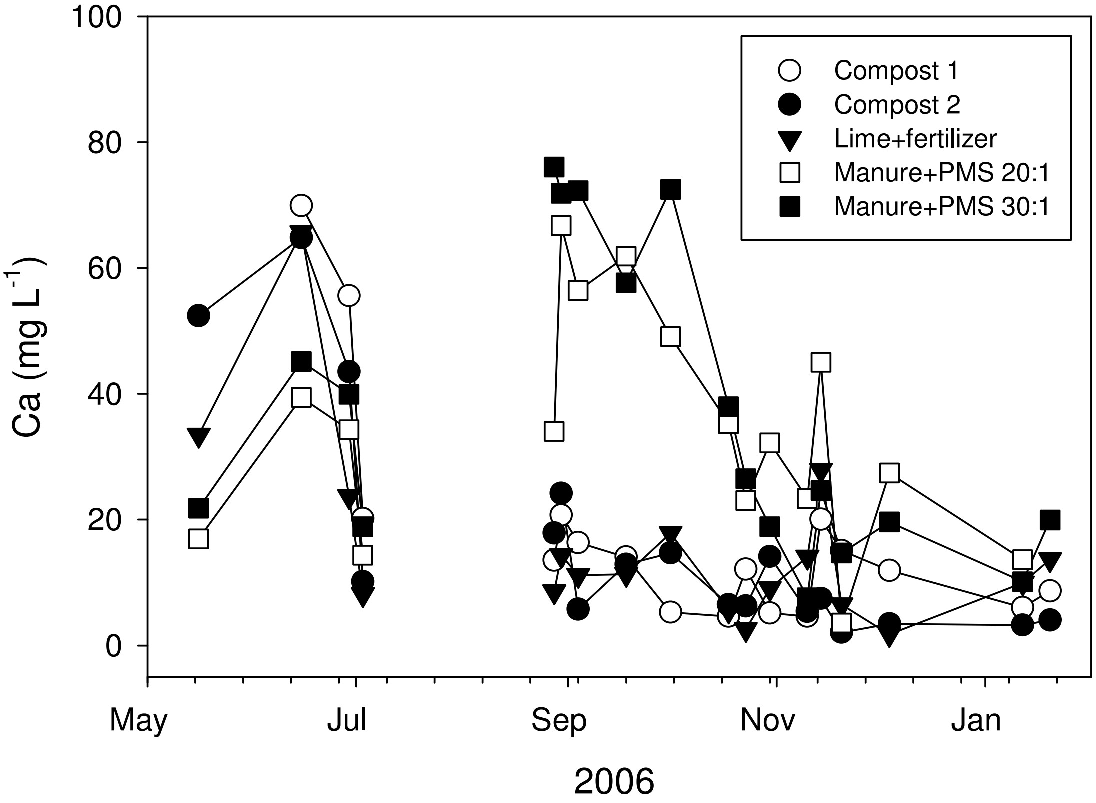


Figure 14. Calcium leached by treatment during the first year of the field experiment. No data available for August 2006.

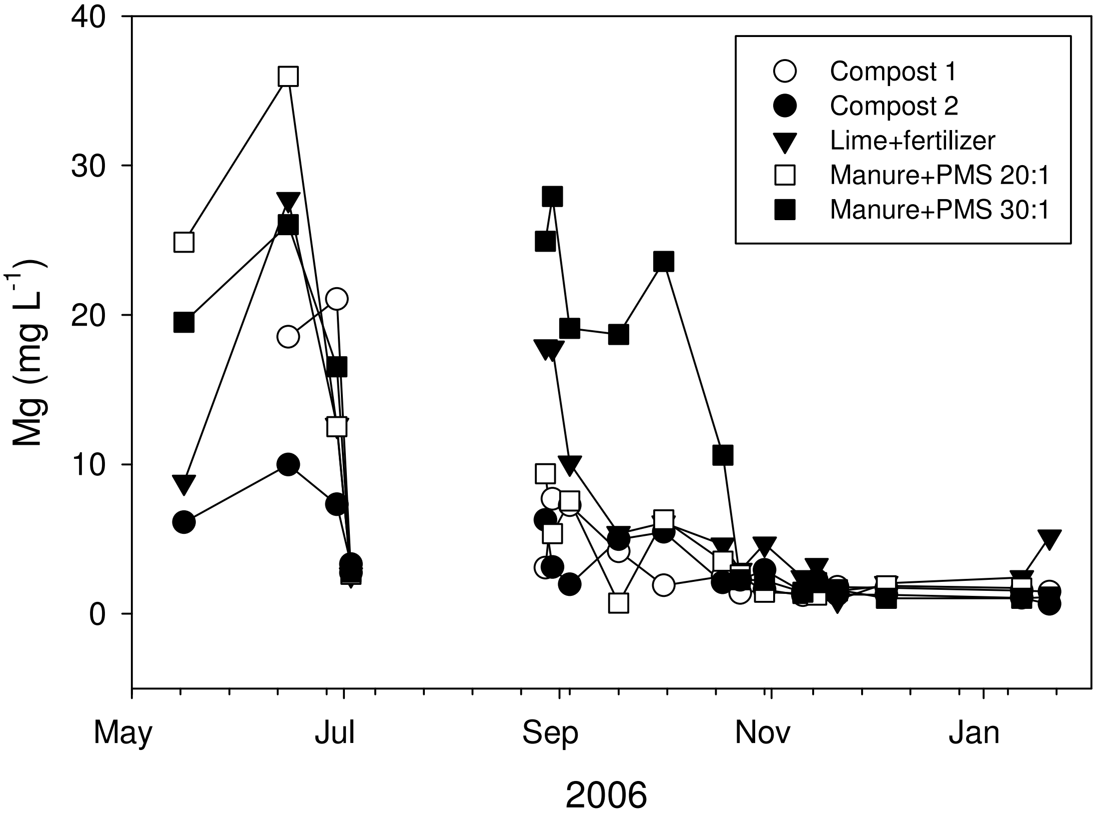


Figure 15. Magnesium leached by treatment during the first year of the field experiment. No data available for August 2006.

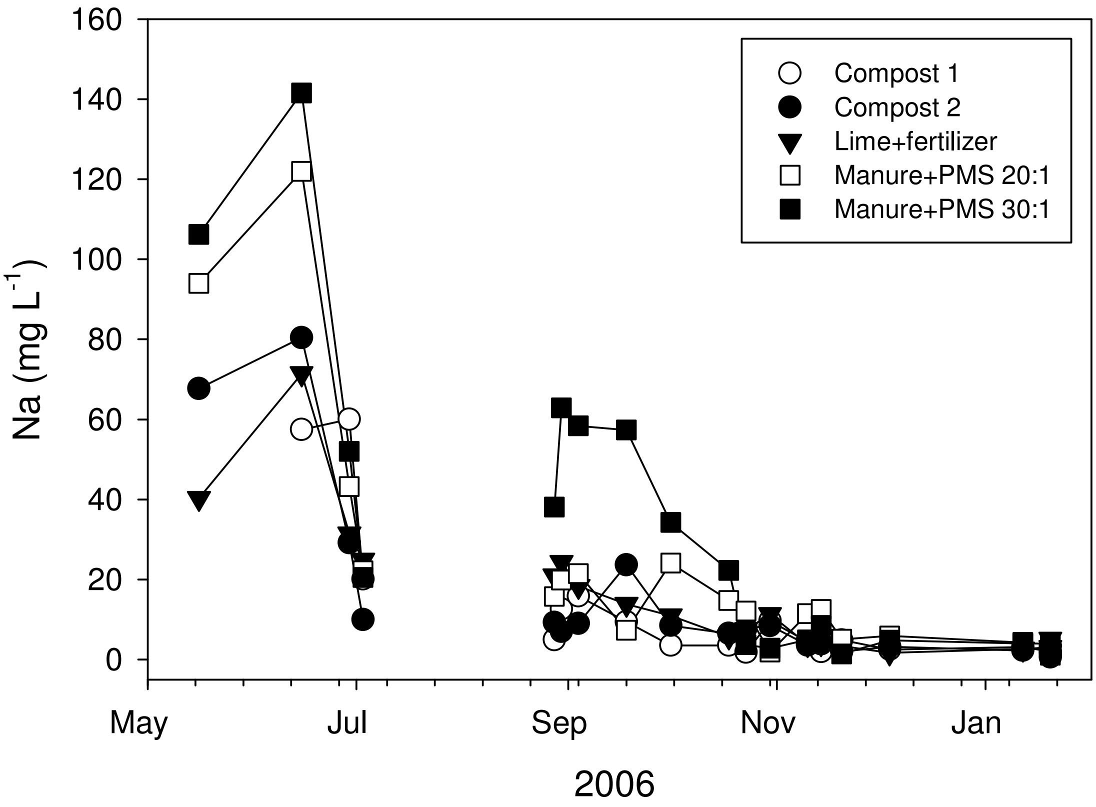


Figure 16. Sodium in leachate by treatment during the first year of the field experiment. No data available for August 2006.

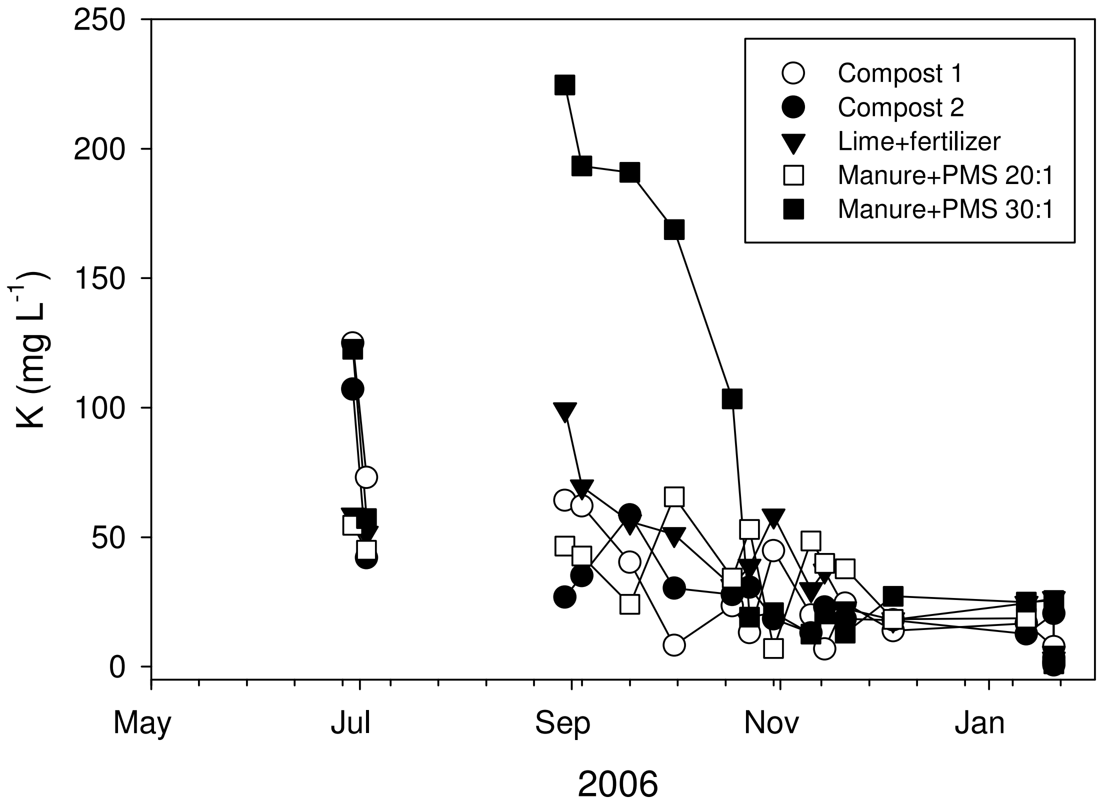


Figure 17. Potassium in leachate by treatment during the first year of the field experiment. No data available in May, June and August 2006.

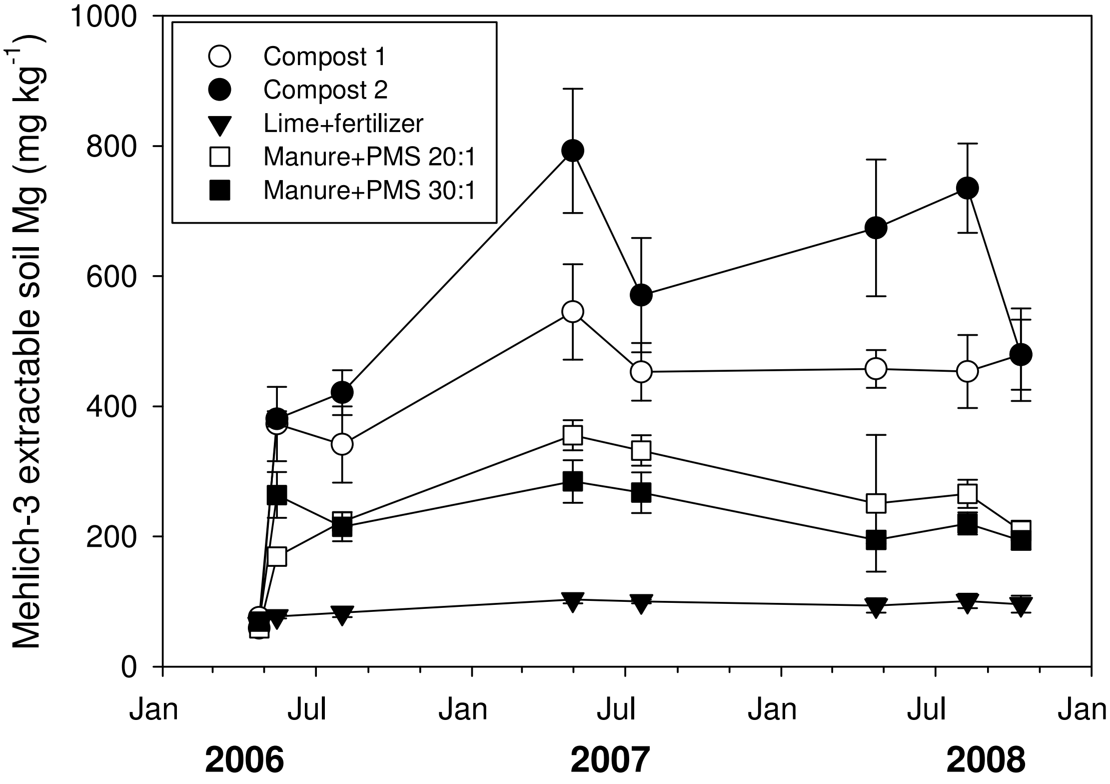


Figure 18. Mehlich-3 extractable soil Mg by treatment over three years.

Table 1. Metal concentrations in leachate by treatment for two selected sampling dates (June and August 2006) in the field experiment.

	Analyte						
	Cd	Cu	Cr	Mo	Ni	Pb	Zn
	mg L^{-1}						
June 2006							
Lime+fertilizer	0.00126	0.0185	—†	—	0.0341	—	0.0103
Compost 1	0.00226	0.0797	—	0.0104	0.0681	0.322	0.284
Compost 2	0.00215	0.0196	—	—	0.0948	—	0.254
Manure+PMS 20:1	—	0.225	—	0.0167	0.229	0.0179	0.190
Manure+PMS 30:1	—	0.145	—	0.0128	0.159	0.00768	0.826
August 2006							
Lime+fertilizer	0.00303	0.00684	—	0.00340	0.0225		0.0363
Compost 1	0.00294	0.0147	—	0.00518	0.0123	0.00346	0.00904
Compost 2	0.00335	0.0171	—	0.00643	0.0121	—	0.00515
Manure+PMS 20:1	0.00295	0.0310	—	0.0107	0.0210	—	0.0197
Manure+PMS 30:1	0.00375	0.0263	—	0.0129	0.0294	0.00661	0.0637

† Below detection limit