CHARACTERIZATION OF NUTRIENT RELEASE AND GREENHOUSE GAS EMISSION FROM CHERNOZEMIC SOILS AMENDED WITH ANAEROBICALLY DIGESTED CATTLE MANURE

BY

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ABSTRACT

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Characterization of Nutrient Release and Greenhouse Gas Emission from Chernozemic Soils Amended with Anaerobically Digested Feedlot Manure.
Major Professor: Dr. Francis Zvomuya.

Understanding the nutrient release patterns of an organic amendment following its land-application is important for both agronomic and environmental reasons. Presented here are findings from two laboratory incubation studies and one growth room bioassay of forage barley (*Hordeum vulgare* L.) investigating nitrogen (N) and phosphorus (P) release, and nitrous oxide (N_2O) emission from a Dark Brown and a Black Chernozem amended with anaerobically digested beef cattle feedlot manure (ADM). The ADM is a nutrient-rich substrate which is cogenerated during anaerobic digestion of livestock manure to produce biogas. Solid and liquid fractions of the ADM are applied to cropland at rates used for raw, non-digested manure since scientific information on nutrient release from ADM is lacking. In all three studies, soils were amended with raw beef cattle feedlot manure, the separated solids fraction of ADM (SS), and pelletized SS (PSS) at rates calculated to supply 400 and 800 mg N kg^{-1}. Synthetic fertilizer and non-amended (control) treatments were included for comparison. The main hypotheses were that (i) soils amended with SS and PSS emit lower N_2O than soils amended with raw manure, and (ii) SS and PSS release N and P differently from raw manure when these amendments are applied to soils as nutrient sources for crops, due to the biophysicochemical changes which occur during anaerobic digestion. Soils amended with SS had lower cumulative

N_2O emissions (2.4 mg N kg^{-1}) than those amended with manure, while PSS- and manure- amended soils had similar cumulative N_2O emissions (11 mg N kg^{-1}). Incubated manure-, SS-, and PSS-treated soils had variable extents of N immobilization during the 10-wk incubation. However, at the end of the 70-d incubation, manure in both soils and SS in the Black Chernozem resulted in net N mineralization while the SS- and PSS-amended Dark Brown Chernozem and the PSS-amended Black Chernozem had net N immobilization. In contrast, all amendments resulted in net P mineralization throughout the incubation. Apparent N recovery in the bioassay was higher for manure (38%) than SS (23%) and PSS (-10%). Phosphorus apparent recoveries were higher and similar for manure and SS (28%) than PSS (1%). These results show that SS and PSS have lower N supply capacity than raw manure. This may necessitate supplementation of SS and PSS with mineral N fertilizer to ensure an adequate supply of N to crops.

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1. INTRODUCTION

1.1 Concentrated Manure Production: A Management Challenge

Livestock production has become highly concentrated for improved profitability (Beegle et al., 2008) with the result that large quantities of manure are produced in smaller areas (Hofmann and Beaulieu, 2006). In 2001, Canadian livestock produced an estimated 180 million tonnes of manure (Hofmann and Beaulieu, 2006), a quantity similar to the 200 million tonnes of manure produced yearly in the United States (Havlin et al., 2005). Traditionally, manure has been land-applied as a nutrient source for crops and also to improve or maintain soil quality (Johnston, 1994; Larney and Janzen, 1996). However, in confined feeding operations (CFOs), the disposal of manure is often limited to areas close to the CFOs such that manure is often applied at a high frequency and high rates in these areas (Larney et al., 2000; Whalen et al., 2001). Accumulation of nutrients in soils, particularly nitrogen (N) and phosphorus (P), increases the risk of surface and groundwater pollution, especially when manure application is N-based (Eghball, 2002; Eghball, 2002; Hao et al., 2008). With the growing environmental and economic issues associated with manure management, use of anaerobic digestion (Pitts, 2010), along with other manure treatment technologies, such as physical solid/liquid separation and chemical addition (Gungor and Karthikeyan, 2005), is increasing.

1.2 Anaerobic digestion: An Opportunity

Anaerobic digestion plays an important role in the quest for sustainable solutions for the handling and recycling of animal manure and other organic wastes (Holm-Nielsen et al., 2009). In most cases, fresh livestock manure or slurry is fed into a biogas plant and co-digested with digestible organic wastes at thermophilic (53-58°C) and mesophilic (30-40°C) temperatures (Holm-Nielsen et al., 2009) during 12-25 d or a longer time period at psychrophilic (15-20°C) temperatures (Masse et al., 1996).

Similar to composting, anaerobic digestion kills pathogens and reduces weed seed viability (Eghball and Power, 1994; Hammac et al., 2007; Rynk, 1992). Losses of C and N during composting contribute towards greenhouse gas (GHG) (methane, CH_4; carbon dioxide, CO_2 and nitrous oxide, N_2O) emissions (Hao et al., 2001), and large quantities of N are also lost via NH_3 volatilization during composting of manure (Eghball et al., 1997). In contrast, anaerobic digestion captures the gaseous C (that is, CO_2 and CH_4) losses and converts them to a renewable energy source (Masse et al., 1996), with a nutrient-rich substrate, fibre, and reusable water as co-products (Holm-Nielsen et al., 2009).

The anaerobically digested manure (ADM) co-generated during biogas production from anaerobic digestion of livestock manure is a nutrient-rich substrate which can be transported back to CFOs and other livestock farms which supply the biogas plant, but only in amounts consistent with regulations on nutrient loading (Holm-Nielsen et al., 2009) to prevent nutrient accumulation in the receiving soils. Excess ADM can be sold or given to crop producers as a source of nutrients for their crops.

Anaerobically digested manure is often separated into solid and liquid fractions that are applied to fields as nutrient sources for crops. Solid-liquid separation enables the partitioning of most of the dissolved N to the liquid fraction while the less available N along with a larger fraction of total P remains in the solid fraction (Moller et al., 2006). The result is a liquid fraction with a high N:P ratio that can be applied close to CFOs with lower potential for P accumulation than raw manure or ADM slurry. The solid fraction of ADM (SS) with less available N than raw manure and usually higher P content than manure can be hauled over longer distances than the liquid fraction, allowing nutrient export to nutrient deficient areas (Holm-Nielsen et al., 2009). To increase the haulage distance of nutrients away from CFOs, manure or ADM can be pelletized. Pelletization reduces moisture content, improves the ease of handling and storage, and increases the economic haulage distance of manure nutrients (Hammac et al., 2007).

1.3 Nutrient Dynamics Following Application of Organic Amendments

In order to apply manure or other organic amendments to meet crop nutrient requirements, knowledge of the amount of nutrients mineralized following application is needed (Eghball et al., 2002). The precise measurement of nutrient mineralization, especially N, is desirable if ADM is to be used efficiently without loss of yield and increased risk of environmental pollution (Rees, 1989). In western Canada, ADM is applied to cropland at the same rates as manure despite possible changes in nutrient availability due to anaerobic digestion. This is based on the assumption that any changes occurring during digestion will not significantly affect nutrient availability. Manure processing, through, for example, composting (Eghball et al., 2002) and pelletization (Hammac et al., 2007), alters nutrient mineralization when the processed product is applied to soil. Therefore, it is likely that raw manure and ADM have different N and P mineralization characteristics.

Application of N fertilizers and organic amendments to soils contributes directly to the increase in N_2O emissions from agricultural soils through stimulation of nitrification and denitrification (Bouwman et al., 2002; Mosier et al., 1998; Rochette et al., 2008).

Nitrous oxide in the troposphere absorbs thermal radiation and thus contributes to the GHG effect (Mosier et al., 1998), with 300 times greater global warming potential than CO_2 (Intergovernmental Panel on Climate Change, 2001). While N_2O emissions from agricultural soils amended with N fertilizers and raw, composted or anaerobically stored manure and other organic amendments have been reported (Akiyama et al., 2004; Burger and Venterea, 2008; Paul et al., 1993), information on N_2O emissions from soils amended with ADM is generally lacking. It is likely that due to the biophysicochemical changes occurring during anaerobic digestion, N_2O emissions from ADM application may be lower than those from raw manure.

Controlled-environment experiments, such as laboratory incubations and growth room bioassays, eliminate variations in nutrient dynamics due to temperature and moisture, the main environmental drivers of microbial activity and hence mineralization and N_2O emission (Andersen and Jensen, 2001; Linn and Doran, 1984). Mineral N or P released during aerobic laboratory incubation of soil cores provides a measurement of net mineralization or immobilization since alternative inorganic N or P pathways, such as plant uptake and leaching, are eliminated (Rees, 1989). Growth room studies enable approximation of competition between soil microorganisms and plant roots for amendment N (Chadwick et al., 2000; Helgason et al., 2007) and P (Hammac et al., 2007; Zvomuya et al., 2006) by crop growth and continuous removal of inorganic N and P from the soil by plant roots. Because soil moisture and temperature conditions are optimized, incubation and growth room studies give an insight into the nutrient dynamics following amendment application to soil and are useful as a measure of potential nutrient mineralization (Stanford, 1982). Given this background, the objectives of this thesis were to:

(a) Determine N_2O emissions from agricultural soils amended with ADM relative to those amended with raw, non-digested manure (Chapter 2),

(b) Characterize the N and P release potentials of SS and pelletized SS (PSS) vs. raw feedlot manure following application to two contrasting soils (Chapter 3),

(c) Characterize nutrient release from SS and PSS vs. raw feedlot manure through N and P uptake by forage barley (*Hordeum vulgare* L.) (Chapter 4), and

(d) Determine effects of N rate on N_2O emission, N and P release, and plant N and P uptake (Chapters 2, 3 and 4, respectively).

These objectives were achieved using controlled environment experiments consisting of two 10-wk incubation studies and a 30-wk growth room bioassay of barley. Two soils (a Dark Brown and a Black Chernozem), four amendments [raw beef cattle feedlot manure, SS, PSS, and a synthetic fertilizer (urea plus monoammonium phosphate, UMP)], and two amendment rates (400 and 800 mg N kg^{-1}) were used in all three experiments. Plate 1 of Appendix I shows a picture of the raw manure, SS and PSS.

Chapter 2 of this thesis details the findings from a 10-wk incubation study, which quantified and compared N_2O emission from soils amended with SS and PSS vs. raw feedlot manure. Chapter 3 (based on findings from an incubation study similar to that in Chapter 2) reports on the N and P mineralization characteristics of raw feedlot manure vs. the two ADM forms. Chapter 4 reports on N and P mineralization in soils amended with the two ADM forms vs. raw manure as shown by plant N and P uptake. Chapter 5 is a general synthesis of the findings from the controlled-environment experiments described above. This thesis as a whole is an effort towards better understanding of nutrient dynamics in agricultural soils amended with ADM, a prerequisite for sustainable use of the ADM as a nutrient source for crops.

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2. NITROUS OXIDE EMISSIONS FROM CHERNOZEMIC SOILS AMENDED WITH ANAEROBICALLY DIGESTED BEEF CATTLE FEEDLOT MANURE: A LABORATORY STUDY[1]

2.1 Abstract

Biogas production from beef cattle (*Bos taurus*) feedlot manure results in the co-generation of anaerobically digested manure (ADM), a nutrient-rich substrate, the solid fraction (SS) of which is often applied to cropland. Application of SS to cropland may result in lower nitrous oxide (N_2O) emissions than raw manure due to biochemical changes occurring during anaerobic digestion. This hypothesis was tested using a laboratory incubation study in which N_2O fluxes from two Alberta soils [a Dark Brown Chernozem clay loam (Typic Haploboroll) and a Black Chernozem silty clay (Typic Haplocryoll)] amended with raw beef cattle feedlot manure, SS, pelletized SS (PSS), and urea + mono-ammonium phosphate (UMP) were measured over a 10-wk period. Amendments were applied at rates corresponding to 400 and 800 mg N kg^{-1}, with a 0 N control included for comparison. Amended soils were maintained near 70% of field capacity moisture content and incubated for 10 wk at 22°C. Gas samples were collected 0, 3 and 7 d after the start of the incubation and at weekly intervals thereafter for N_2O and CO_2 analysis by gas chromatography. Analysis of variance (ANOVA) showed that N_2O

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[1] Chiyoka, W.L.[1], X. Hao[2], F. Zvomuya[1], and X. Li[3]. 2011. Nitrous oxide emissions from Chernozemic soils amended with anaerobically digested beef cattle feedlot manure: A laboratory study. Anim. Feed Sci. Tech. (in press). [1]University of Manitoba; [2]Agriculture and Agri-Food Canada Lethbridge Research Centre, Lethbridge, AB; [3]Highmark Renewables Inc., Edmonton, AB.

fluxes differed significantly among amendments with the differences dependent on soil, amendment application rate, and time.

Across amendment rates, mean cumulative emissions from SS- (2.38 mg N kg^{-1}) and UMP-amended soils (0.59 mg N kg^{-1}) were not significantly different, but both were significantly lower than emissions from PSS-amended soils (10.7 mg N kg^{-1}) and those from the higher manure rate (15.6 mg N kg^{-1}). The high N_2O emissions from PSS-amended soils were likely due to the concentrated microsites of N in pellets. The difference in cumulative N_2O emissions between PSS and SS was only significant in the Black Chernozem. Microbial activity as indicated by cumulative CO_2 fluxes was highest in SS-amended (6.7 g CO_2-C kg^{-1}) and lowest in UMP-amended soils (0.14 g CO_2-C kg^{-1}). Post-incubation net mineral N content was greatest in manure-amended soils (144 mg N kg^{-1}), marginal in SS-amended soils (23 mg N kg^{-1}), and lowest in PSS-amended soils that had net N immobilization (-67 mg N kg^{-1}) due to low available N content in the pellets. These laboratory results suggest that field application of SS may reduce N_2O emissions relative to raw manure.

2.2 Introduction

Anaerobic digestion (AD) of livestock manure produces beneficial value-added products. Although AD does not reduce nutrient loading from manure application, the process creates alternative nutrient management opportunities and significantly reduces odor, pathogens, and viability of weed seeds that pass through animal digestive systems (Masse et al., 1996; Climate Solutions, 2002; Moser, 2007). Biogas is the primary product from the AD process, with anaerobically digested manure (ADM) comprising water, fibre and nutrients as a secondary product. The nutrient-rich ADM is often separated into solid and liquid fractions that are applied to cropland as nutrient sources for crops.

Application of synthetic N fertilizers, manure, and other organic amendments to cropland contributes directly to N_2O emission through stimulation of nitrification and denitrification (Mosier et al., 1998; Bouwman et al., 2002; Smith et al., 2007; Rochette et al., 2008; Schils et al., 2008). Nitrous oxide is a potent greenhouse gas that has also been linked to depletion of the ozone layer (Crutzen, 1981; Bouwman, 1990). Several studies have reported N_2O emissions from agricultural soils amended with synthetic fertilizers and raw, composted or anaerobically stored manure and other organic amendments in laboratory incubation experiments (Paul et al., 1993; Akiyama et al., 2004; Burger and Venterea, 2008) and under field conditions (Rochette et al., 2008; Schils et al., 2008). However, information on N_2O emissions from soils amended with ADM is generally lacking.

The Intergovernmental Panel on Climate Change (2006) assigns an N_2O emission factor of 1% of N applied as mineral fertilizers and manure; however, information on the applicability of this factor to anaerobically digested manure is currently lacking. Physical and biochemical changes during anaerobic digestion of manure may alter N_2O and CO_2 emissions associated with land application of ADM relative to raw manure. Given the growing interest in anaerobic digestion for generating biogas and managing large volumes of manure from concentrated animal feeding operations (Climate Solutions, 2002; Moser, 2007), there is a need for accurate data on N_2O emissions from land application of the resulting ADM. Such information will in turn assist governments in the preparation of accurate annual greenhouse gas inventories for the agriculture sector.

The overall objective of this study was to determine N_2O emissions from agricultural soils amended with ADM relative to those amended with raw, non-digested manure. It was hypothesized that, because of biophysicochemical changes occurring during anaerobic digestion, N_2O emission from soils amended with ADM would be lower than that from soils amended with raw, non-digested manure.

2.3 Materials and Methods

2.3.1 Soils and organic amendments

A microcosm incubation experiment was conducted using two soils (0-15 cm) from sites in Alberta, Canada. The soils were a Dark Brown Chernozem (fine-loamy, mixed, Typic Haploboroll) from a site near Lethbridge (49°42'N; 112°47'W) and a Black Chernozem (silty clay Typic Haplocryoll) from a site near St. Albert, (53°42'N; 113°47'W). The soils were air-dried, sieved through a 4-mm sieve and stored at room temperature until the start of the experiment.

Air-dried soil samples (< 2mm) were analyzed for particle size distribution (Gee and Bauder, 1986), pH and EC (1:2 soil: water ratio), and total C and N concentrations (NS-2000 Nitrogen Analyzer, Leco Corporation, St. Joseph, MI) (Table 2.1). Soil inorganic N concentration was determined by the phenate method (Searle, 1984) using a Model AA3 autoanalyzer (Bran+Luebbe, Nordersted, Germany) following extraction of 5 g of soil with 25 mL of 2 M KCl (Keeney and Nelson, 1982). Soil moisture content at field capacity (FC) was determined under a suction of 30 kPa using a 500 kPa pressure plate moisture extractor (Model 1600, Soil Moisture Equipment Co., Santa Barbara, CA).

Table 2.1 Selected initial physical and chemical properties of the Dark Brown and Black Chernozemic soils used in the microcosm experiment.

Property[†]	Dark Brown	Black
pH	6.3	6.3
Total N (g kg^{-1})	2.2	7.1
NH_4^+-N (mg kg^{-1})	5.0	8.0
NO_3^--N (mg kg^{-1})	8.0	34
Organic C (g kg^{-1})	31	91
Clay (g kg^{-1})	294	450
Silt (g kg^{-1})	271	480
Sand (g kg^{-1})	435	70
Field capacity moisture content (g kg^{-1})	283	450

[†] Soil pH measured on fresh weight basis; Total N, NH_4^+-N, NO_3^--N, and organic C expressed on a dry weight basis.

Table 2.2 Initial properties of raw and anaerobically-digested manure forms applied to Black and Dark Brown Chernozems.

Property[†]	Raw manure	SS[‡]	PSS[§]
Moisture content (g kg^{-1})	628	745	91
pH	7.8	8.7	7.6
Electrical conductivity (dS m^{-1})	10.8	4.49	6.78
Total C (g kg^{-1})	274	389	391
Total N (g kg^{-1})	19	16	12
C:N ratio	14	24	33
NH_4^+-N (mg kg^{-1})	3493	2433	217

[†] Moisture content, pH, and electrical conductivity on fresh weight basis; total N, total C, NH_4^+-N, and C:N ratio on a dry weight basis.
[‡] Separated solids from anaerobically digested manure slurry.
[§] Pelletized separated solids.

Freeze-dried organic amendments (Table 2.2) were analyzed as described above for soil, except for available N, which was determined in fresh samples. The pellets were cylindrical and approximately 9 mm long with a diameter of 4 mm and density of 1.2 Mg/m^3. Pelletization of livestock manure is a common practice mainly because it (i) reduces moisture content and increases economic hauling distance of manure; (ii) reduces odor; and (iii) improves the ease of manure storage and handling (Hammac et al., 2007). Pelletization of SS aims to achieve these same benefits.

2.3.2 Microcosm setup

Raw manure, SS, PSS, and a synthetic fertilizer blend (urea plus monoammonium phosphate, UMP) were applied as sub-treatments to soils (main plots) at rates (sub-sub-plots) corresponding to 0, 400, and 800 mg N kg^{-1}. The 400 mg N kg^{-1} rate corresponds to the 100 kg N ha^{-1}, the standard recommendation for silage barley (*Hordeum vulgare* L.) production in western Canada. All treatments were replicated three times.

Prior to amendment application, pre-wetted soils (<70% FC) were placed in the incubator for 48 h to stabilize soil conditions at 22°C and reduce N_2O peaks due to re-wetting effects (Davidson, 1992; Flessa and Beese, 2000; Mkhabela et al., 2006). Soil moisture was then adjusted to 70% FC with deionized water at the start of incubation. The amount of water added with the organic amendments was considered an amendment effect and not corrected for when soil moisture was adjusted to 70% FC.

Amended soils were packed by hand into polyvinyl chloride cores (5.0 cm high by 5.1 cm diam. with a piece of fabric attached at the base) to bulk densities of 1.1 and 1.0 Mg m^{-3} for the Dark Brown Chernozem and the Black Chernozem, respectively. The amount of amendment added to each soil core was calculated to reflect field-incorporation into the 5-cm soil layer, which is typical of no-till systems for forage barley production. Cores were placed in 1-L wide-mouth sealer jars covered with a piece of parafilm with 5 small pin holes punched to ensure gaseous exchange during the incubation. All jars were randomly arranged inside an incubator (Fisher Scientific IsoTemp Incubator Model 304, Pittsburgh, PA, USA) set to 22°C for 70 d. Plastic containers (32 cm × 23 cm × 6 cm) filled with water were placed in the incubator to increase humidity and reduce moisture loss from the soil cores during incubation. Each core (at 70% FC) was weighed soon after set-up and weekly thereafter to determine and replace water lost by evaporation using deionized water.

2.3.3 Gas sampling and measurement of N_2O and CO_2

Gas samples for N_2O and CO_2 measurements were collected from the cores using a static incubation system (Paul and Beauchamp, 1994; Tenuta and Beauchamp, 2000; Velthof et al., 2003) at 0, 3, and 7 d after the start of incubation and at ~7-d intervals thereafter for the rest of the experiment. At each sampling date, the headspace of each jar was first flushed with air to ensure N_2O and CO_2 were at ambient levels, and then sealed with an aluminum screw cap fitted with a rubber septum for 2 h. After 2 h, an 11-mL sample was taken from the gas accumulated in the headspace of each closed jar, using a 23G1 PrecisionGlide® needle mounted on a 20 mL syringe (BD, Franklin Lakes, NJ) and stored in pre-evacuated 6-mL Exetainer vial (Labco, Buckinghamshire, England). Blank jars were included to correct for background (ambient) N_2O and CO_2 concentrations in the laboratory air. Nitrous oxide and CO_2 concentrations were measured with a gas chromatograph (Varian 3800, Varian Instruments, Walnut Creek, CA) equipped with separate detectors for N_2O (electron capture detector; carrier gas 10% methane and 90% argon) and CO_2 (thermal conductivity detector; carrier gas helium). Background N_2O and CO_2 concentrations were subtracted from the measurements prior to data analysis.

2.3.4 Post-incubation soil analysis

At the end of the incubation, soil samples from the microcosms were analyzed for pH (soil: water ratio of 1:2) and for inorganic N (NO_2^-, NO_3^- + NH_4^+) as described for initial soil analysis. Net treatment effects on soil mineral N contents were assessed after subtracting inorganic N concentrations of control soils from those of amended soils.

2.3.5 Calculation of N_2O and CO_2 fluxes

Nitrous oxide and CO_2 fluxes (μg N kg^{-1} h^{-1}) were calculated from the rates of accumulation of these gases in the headspace of each jar, assuming a linear increase in gas concentration with time (Velthof and Oenema, 1995; Velthof et al., 2003). Dissolved N_2O-N was calculated according to Moraghan and Buresh (1977) and added to headspace N_2O concentration to give total N_2O flux. Cumulative N_2O and CO_2 emissions were calculated by integrating gas fluxes over the incubation period. For assessing net amendment and rate effects, the cumulative emissions of each gas from non-amended soils were subtracted from those for amended soils.

2.3.6 Statistical analysis

All data, except cumulative emissions and post-incubation inorganic soil N, were analyzed using the MIXED procedure for repeated measures in SAS (Littell et al., 1998; SAS Institute, 2008) with time as the repeated factor. Soil, amendment, and rate were fixed effects while replication and interaction of replication with fixed effects were considered as random effects. The spatial power [SP(POW)] covariance structure was used in the model for these repeated measures data in which the sampling time intervals were unequal. Nitrous oxide and CO_2 fluxes were $\log_e$-transformed prior to analysis to meet assumptions of normality (Shapiro Wilk's test) and homoscedacity (Levene's test). Amendment and rate effects on cumulative N_2O and CO_2 emissions and on post-incubation inorganic soil N content were assessed using the MIXED procedure. Treatment differences were considered significant if $P < 0.05$ using the Tukey-Kramer method.

2.4 Results

2.4.1 Nitrous oxide fluxes

Nitrous oxide fluxes differed significantly ($P < 0.0001$) among amendments; however, amendment differences varied with soil, amendment rate, and time, as indicated by the significant ($P < 0.0001$) four-way interaction (Table 2.3). At the 400 mg N kg^{-1} rate, N_2O fluxes were highest for raw manure at nearly all sampling dates in the Dark Brown Chernozem (Fig. 2.1a), whereas PSS resulted in the highest fluxes at all but the first sampling date in the Black Chernozem (Fig. 2.1c). The pattern was different at the 800 mg N kg^{-1} rate where the highest N_2O fluxes in the Dark Brown Chernozem were associated with UMP but only after day 20 (Fig. 2.1b), while N_2O fluxes from the UMP-amended Black Chernozem were lower than those from manure, SS, and PSS during the sampling period between days 3 and 49 (Fig. 2.1d).

Compared with raw manure, SS application resulted in lower fluxes in the Dark Brown Chernozem except on days 0, 3, 42 and 49 at the 400 mg N kg^{-1} rate (Fig. 2.1a) and days 0, 3, and 42 through 70 at the 800 mg N kg^{-1} rate (Fig. 2.1b) when differences between the two amendments were not significant ($P > 0.05$). By contrast, in the Black Chernozem, differences in N_2O flux between SS and raw manure were significant only on day 3 at both amendment rates. Nitrous oxide fluxes from PSS and SS treatments were similar and differed in the same way from manure effects at both amendment rates, except on day 7 when the flux from the PSS treatment at the 800 mg N kg^{-1} rate was significantly higher than that from SS and similar to that from manure application. Also, for the Black Chernozem, PSS resulted in significantly higher N_2O fluxes than manure and SS on all but days 0 (flux lower for PSS), 3 and 7 (PSS similar to manure), and 21 (PSS similar to SS) at the 400 mg N kg^{-1} rate, whereas, at the 800 mg N kg^{-1} rate, significant differences were detected only on days 0 (flux lower for PSS), 3 (flux higher for PSS than SS), and 7 (lower for PSS than manure).

Table 2.3 Soil, amendment, and rate effects on nitrous oxide (N_2O) and carbon dioxide (CO_2) fluxes from, and net inorganic N accumulation in Black and Dark Brown Chernozemic soils.

Effect	Flux		Cumulative losses		Soil inorganic N†
	N_2O (μg N kg^{-1} h^{-1})	CO_2 (mg C kg^{-1} h^{-1})	N_2O (mg N kg^{-1})	CO_2 (g C kg^{-1})	(mg N kg^{-1})
Soil					
Dark Brown	0.50	2.32	4.35	4.02	92
Black	0.90	2.62	7.78	3.86	91
Amendment					
Manure	1.12	2.81	10.6	5.60	144
SS	0.50	3.18	2.38	6.73	23
PSS	0.87	2.55	10.7	3.29	-67
UMP	0.39	1.62	0.589	0.14	264
Rate (kg N ha^{-1})					
0	0.12	1.57	-	-	-
400	0.87	2.68	4.03	2.65	59
800	1.46	3.56	8.1	5.24	124
			P-value		
Soil (S)	<0.0001	<0.0001	0.0002	0.16	0.86
Amendment (A)	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
Rate (R)	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
Time (T)	<0.0001	<0.0001	-	-	-
S × A	<0.0001	0.06	<0.0001	0.015	<0.0001
S × R	<0.0001	0.001	0.65	0.51	0.94
A × R	<0.0001	<0.0001	0.002	0.0003	<0.0001
S × A × R	<0.0001	0.87	0.68	0.98	0.003
S × T	<0.0001	<0.0001	-	-	-
A × T	<0.0001	<0.0001	-	-	-
S × A × T	<0.0001	<0.0001	-	-	-
R × T	<0.0001	<0.0001	-	-	-
S × R × T	<0.0001	0.55	-	-	-
A × R× T	<0.0001	<0.0001	-	-	-
S × A × R × T	<0.0001	0.01	-	-	-

† Inorganic N, sum of NH_4^+, NO_3^- and NO_2^- concentrations.

‡ Time main effects not shown for N_2O and CO_2 fluxes because of the multiple sampling times. Four-way interactions including Time as a factor are graphed for N_2O and CO_2 fluxes.

In general, all treatments increased N_2O flux compared with the unamended control, with the exception of UMP, which, at the 400 mg N kg^{-1} rate in the Dark Brown Chernozem, resulted in a significant increase only on day 3. The differences in N_2O flux between amended and unamended soils also decreased with time as increasing amounts of the readily denitrifiable amendment N were lost. On day 0, at both amendment rates, N_2O fluxes from soils amended with UMP were not significantly different from those for the unamended Dark Brown Chernozem, but significantly higher than those from the unamended Black Chernozem. Fluxes from PSS, on the other hand, were similar to those for the control (unamended soil) regardless of soil type or rate.

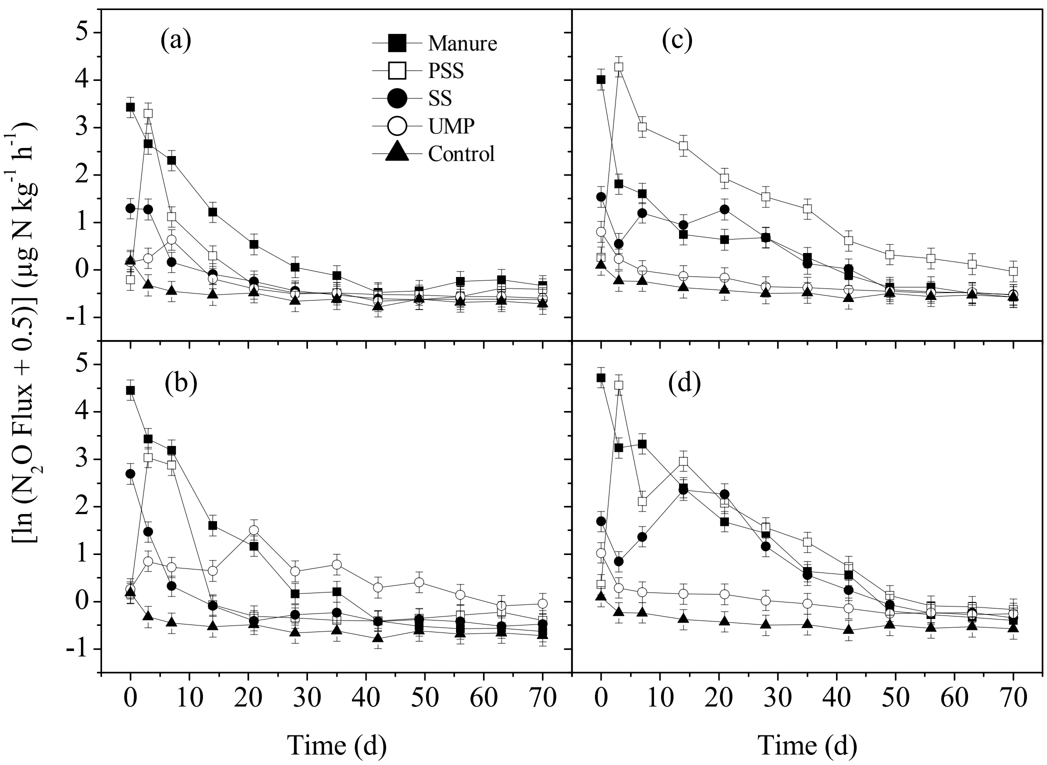

Figure 2.1 Temporal changes in nitrous oxide (N_2O) fluxes during microcosm incubation as affected by soil type, amendment type, and rate of application. Manure, raw beef cattle feedlot manure; control, non-amended soil; SS, separated solids from anaerobic digestion of beef cattle feedlot manure, PSS (pelletized SS), and UMP (urea plus monoammonium phosphate) applied at 400 and 800 mg N kg^{-1} rates. Fluxes are given for the Dark Brown Chernozem at the 400 mg N kg^{-1} rate (a) and 800 mg N kg^{-1} (b), and in (c) and (d) fluxes from the 400 and 800 mg N kg^{-1} rates, respectively for the Black Chernozem.

2.4.2 Cumulative N_2O emissions

There were significant soil × amendment and amendment × rate interactions for cumulative N_2O losses (Table 2.3). The soil × amendment interaction was due to the difference in N_2O losses between PSS and SS, which was significant for the Black Chernozem (16.2 mg N kg^{-1} for PSS vs. 3.6 mg N kg^{-1} for SS) but non-significant for the Dark Brown Chernozem (5.2 vs. 1.1 mg N kg^{-1}) (Fig. 2.2). In both soils, N_2O losses were similar for manure (mean 10.6 mg N kg^{-1}) and PSS (10.7 mg N kg^{-1}) and for SS (2.4 mg N kg^{-1}) and UMP (0.6 mg N kg^{-1}). Cumulative losses from each amendment did not differ significantly between the two soils.

Although cumulative N_2O emissions, averaged across the soils, were higher at the 800 mg N kg^{-1} than at the 400 mg N kg^{-1} rate, the difference between the two amendment rates was only significant in manure-amended soils (5.6 mg N kg^{-1} at the 400 mg N kg^{-1} rate vs. 15.6 mg N kg^{-1} at the 800 mg N kg^{-1} rate) (Fig. 2.3). Across amendment rates, cumulative emissions from SS- (mean 2.38 mg N kg^{-1}) and UMP-amended soils (mean 0.59 mg N kg^{-1}) were not significantly different, but both were significantly lower than emissions from PSS-amended soils (mean 10.7 mg N kg^{-1}) and those from the higher manure rate (15.6 mg N kg^{-1}).

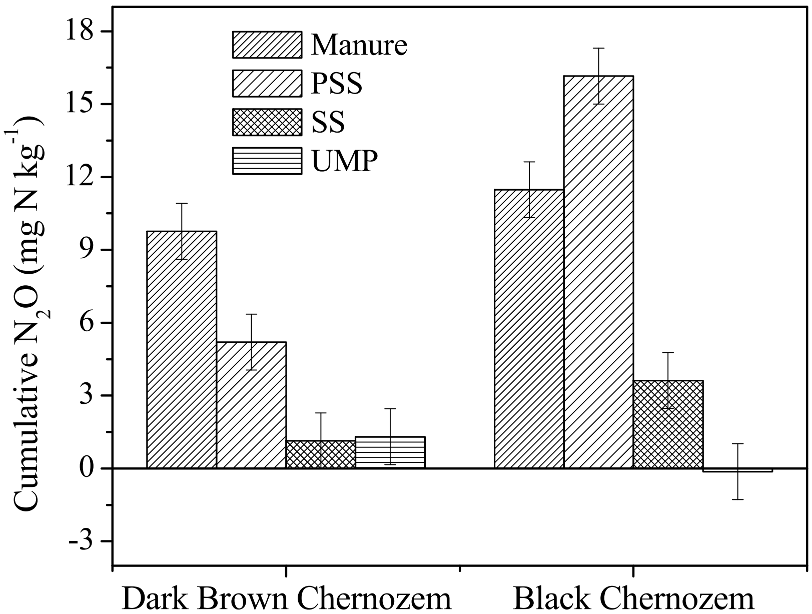

Figure 2.2 Cumulative N_2O emissions from Chernozemic soils amended with raw beef cattle feedlot manure, SS (separated solids from anaerobic digestion of beef cattle feedlot manure), PSS (pelletized SS), and UMP (urea plus monoammonium phosphate) applied at 400 and 800 mg N kg^{-1} rates due to interaction between soil and amendment. Cumulative losses shown are the difference in cumulative N_2O emission between amended and non-amended soils.

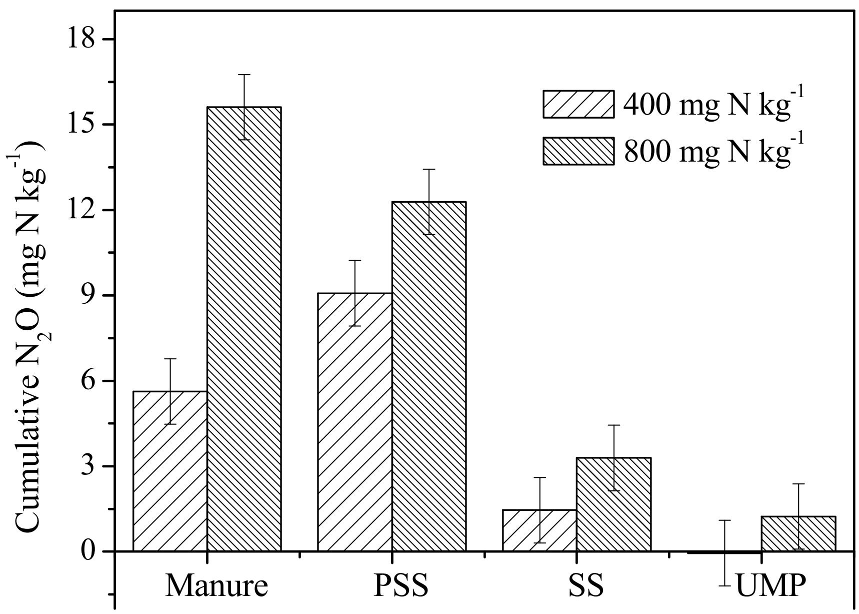

Figure 2.3 Cumulative N_2O emissions from Chernozemic soils amended with raw beef cattle feedlot manure, SS (separated solids from anaerobic digestion of beef cattle feedlot manure), PSS (pelletized SS), and UMP (urea plus monoammonium phosphate) applied at 400 and 800 mg N kg^{-1} rates due to interaction between amendment and rate. Cumulative losses shown are the difference in cumulative N_2O emission between amended and non-amended soils.

2.4.3 Carbon dioxide fluxes

Similarly to N_2O fluxes, the soil × amendment × rate × time interaction was significant for CO_2 fluxes (Table 2.3). In both soils and at both rates, CO_2 fluxes were highest in soils amended with SS at most sampling times (Fig. 2.4). This was more evident at the 800 mg N kg^{-1} rate at which SS gave the highest CO_2 fluxes during the sampling period from day 3 through day 42 in the Dark Brown Chernozem (Fig. 2.4b) and on all but the first two and the last two sampling dates in the Black Chernozem (Fig. 2.4d). At the 400 mg N kg^{-1} rate, CO_2 fluxes were highest for SS on days 7, 14, and 21 in the Dark Brown Chernozem (Fig. 2.4a) and on 5 of the 12 sampling dates in the Black Chernozem (Fig. 2.4c).

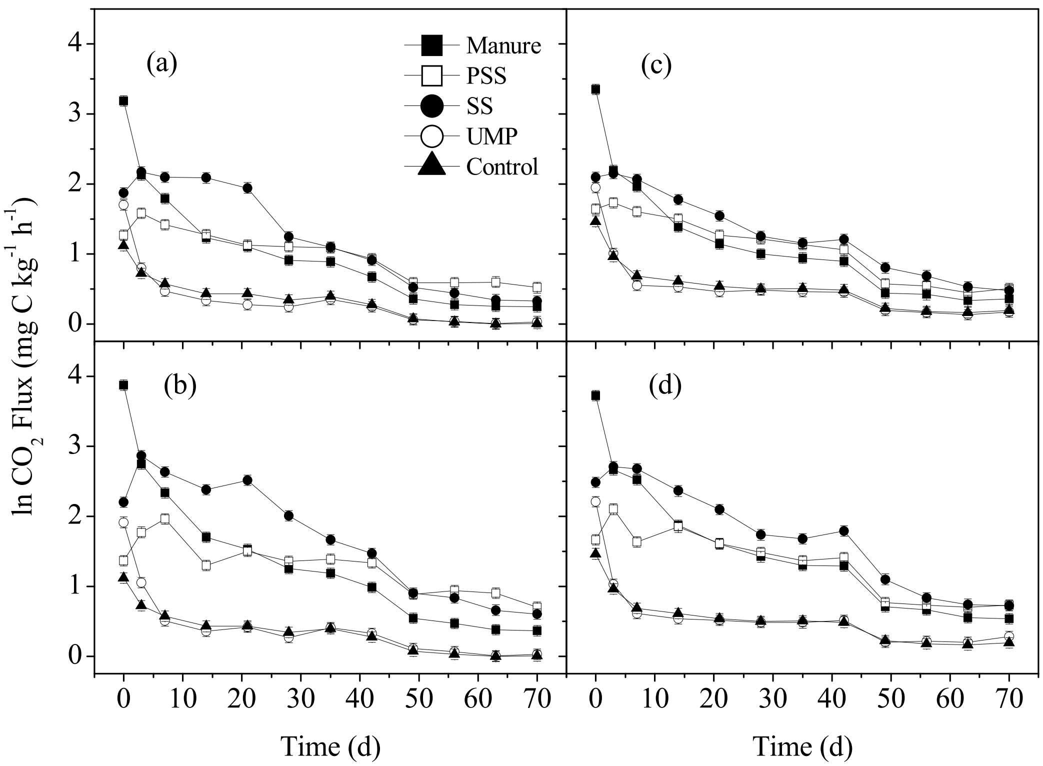

Figure 2.4 Carbon dioxide fluxes from Dark Brown (a and b) and Black (c and d) Chernozemic soils receiving 400 (a and c) and 800 mg N kg^{-1} (b and d) amendment applications of Manure: raw beef cattle feedlot manure; SS: separated solids from anaerobic digestion of beef cattle feedlot manure, PSS: pelletized SS; and UMP: urea plus mono-ammonium phosphate, with a Control: no amendment applied, included.

At all sampling times after day 3, CO_2 fluxes in both soils were higher for SS than manure, except at the 400 mg N kg^{-1} rate in the Dark Brown Chernozem where the fluxes for SS and manure did not differ significantly on or after day 42. Compared with SS, CO_2 fluxes from PSS application were lower during the first 28 d at the 400 mg N kg^{-1} rate and 42 d at the 800 mg N kg^{-1} rate and higher after day 49 at both amendment rates in the Dark Brown Chernozem. By contrast, CO_2 fluxes in the Black Chernozem at both amendment rates were significantly lower for PSS compared with SS at all but the last two sampling times. When compared with manure, CO_2 fluxes from PSS application were lower during the first three sampling dates, except at the 800 mg N kg^{-1} rate in the Dark Brown Chernozem, where the fluxes were lower on day 14 as well. While the CO_2 fluxes did not differ significantly between manure and PSS for the remainder of the sampling dates at both rates in the Black Chernozem, the fluxes were higher for PSS than manure (and SS and UMP) on days 56, 63, and 70 at both rates in the Dark Brown Chernozem.

For both soils and amendment rates, CO_2 fluxes decreased with time for all amendments, although there was an initial increase in flux during the first two sampling dates in soils amended with SS and PSS (Fig. 2.4). In both soils and at both amendment rates, changes in CO_2 flux after day 49 were minimal for all amendments except SS, which appeared to decrease further but at a decreasing rate regardless of soil type or amendment rate. Carbon dioxide emissions from UMP-amended soils were similar to those from non-amended soils irrespective of soil type and amendment rate at all but the first (and second for the 800 mg N kg^{-1} rate in the Dark Brown Chernozem) sampling day(s).

2.4.4 Cumulative CO_2 emissions

Amendment effects on cumulative CO_2 emissions, averaged over rates, varied significantly ($P = 0.02$) with soil (Table 2.3). In both soils, the cumulative emissions were highest and similar for manure and SS (mean 6.2 g CO_2-C for the two amendments) followed by PSS (3.3 g CO_2-C) and UMP (0.1 g CO_2-C) (Fig. 2.5). The interaction in this case was due to the difference in CO_2 emission between manure and PSS being greater in the Dark Brown Chernozem than in the Black Chernozem.

Across the two soils, amendment rate effects on cumulative CO_2 emission differed among the amendments (Table 2.3). Although the cumulative emissions were greater at the 800 than at the 400 mg N kg^{-1} rate for all amendments, the difference between the two rates was greatest in soils amended with SS (4.4 vs. 9.1 g CO_2) and manure (3.7 vs. 7.5 g CO_2) followed by PSS (2.4 vs. 4.2 g CO_2), while emissions from UMP application did not differ significantly between the rates (mean 0.14 g CO_2) (Fig. 2.6).

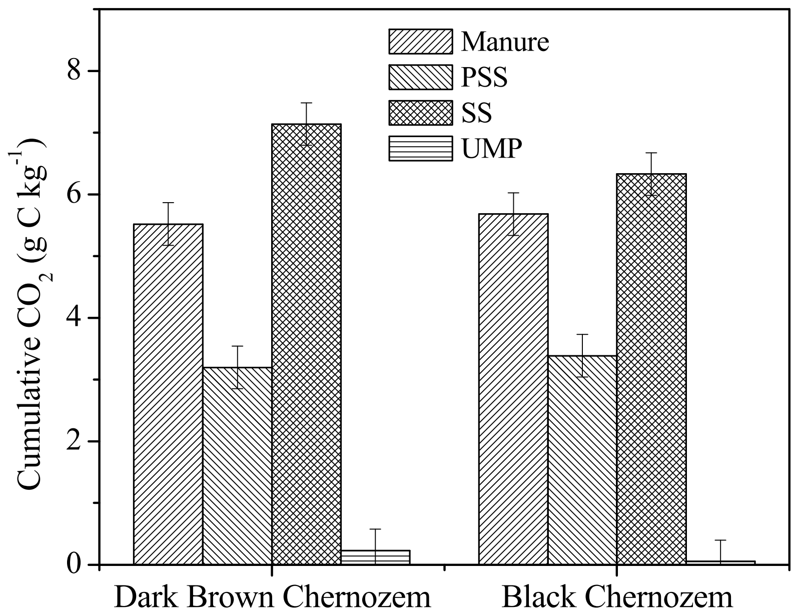

Figure 2.5 Cumulative CO_2 emissions from Chernozemic soils amended with raw beef cattle feedlot manure, SS (separated solids from anaerobic digestion of beef cattle feedlot manure), PSS (pelletized SS), and UMP (urea plus monoammonium phosphate) due to interaction between soil and amendment. Cumulative losses shown are the difference in cumulative N_2O emission between amended and non-amended soils.

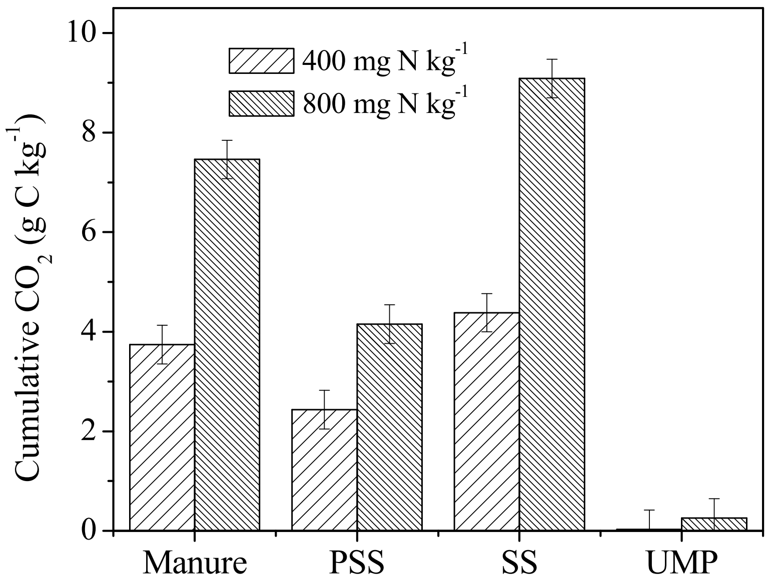

Figure 2.6 Cumulative CO_2 emissions from Chernozemic soils amended with raw beef cattle feedlot manure, SS (separated solids from anaerobic digestion of beef cattle feedlot manure), PSS (pelletized SS), and UMP (urea plus monoammonium phosphate) applied at 400 and 800 mg N kg^{-1} rates due to interaction between amendment and rate. Cumulative losses shown are the difference in cumulative N_2O emission between amended and non-amended soils.

2.4.5 Soil inorganic N concentration

There was a significant ($P = 0.003$) soil × amendment × rate interaction for net (i.e., corrected for background concentrations in the control) soil inorganic N concentration at the end of incubation (Table 2.3). For both soils, inorganic N concentrations were highest with UMP application at the 800 mg N kg^{-1} rate (mean 349 mg N kg^{-1} for the two soils) (Fig. 2.7). The 800 mg N kg^{-1} rate also resulted in higher inorganic N concentrations than the 400 mg N kg^{-1} rate in soils amended with manure and UMP, whereas rate effects were not significant in SS- and PSS-amended soils. In fact, there was a net decrease in inorganic N concentration (immobilization) in soils amended with PSS, while those amended with SS showed only a slight increase. At equivalent amendment rates and for each amendment, inorganic N concentration did not differ significantly between the two soils.

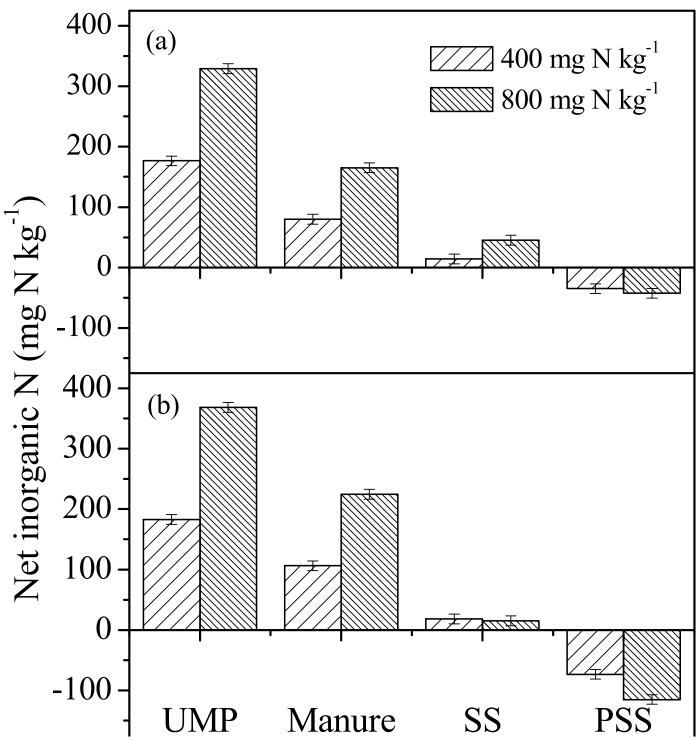

Figure 2.7 Post-incubation net inorganic N contents of the Dark Brown Chernozem (a) and Black Chernozem (b). (For X-axis, Manure: raw beef cattle feedlot manure; SS: separated solids from anaerobic digestion of beef cattle feedlot manure, and PSS: pelletized SS; UMP: urea plus monoammonium phosphate; legends 400 and 800 represent amendment applied at 400 and 800 mg N kg^{-1}; inorganic N contents of Control soils were subtracted from those of amended soils.

2.5 Discussion

2.5.1 Nitrous oxide emissions

Our results show inconsistencies between the Dark Brown Chernozem and Black Chernozem soils with respect to differences in N_2O flux between digested manure forms (i.e., SS and PSS) and manure. While the fluxes were, on average, higher for manure than SS and PSS in the Dark Brown Chernozem, the scenario was different in the Black Chernozem where PSS resulted in higher fluxes (except on the first sampling date) at the 400 mg N kg^{-1} rate while differences among the three amendments were not significant at the higher amendment rate. The higher N_2O fluxes observed in the raw manure-amended Dark Brown Chernozem was likely due to the higher available inorganic N in manure compared with SS and PSS.

It is noteworthy that, despite the increase in soluble inorganic N associated with anaerobic digestion of livestock manure (Loria and Sawyer, 2005), solid/liquid separation of digestate partitions most of the available N into the liquid fraction, leaving the SS with lower available N concentrations than the raw manure (Table 2.2). Nitrous oxide flux in most agricultural soils increases with an increase in available N, which in turn accelerates nitrification and denitrification (Bouwman, 1996; Mosier et al., 1998; Bouwman et al., 2002).

The observed disparities between the two soils were at least in part due to differences in the N and C economy of the soils prior to amendment application. For example, because native (pre-treatment) available N (NH_4^+ plus NO_3^-) concentration (42 mg N kg^{-1}) was higher and the C/N ratio (14) was lower in the Black Chernozem than in the Dark Brown Chernozem (13 mg N kg^{-1} and 22, respectively) (Table 2.1), application of manure, which has higher available N and a lower C/N ratio, would favour a greater increase in N_2O flux in the Dark Brown Chernozem compared with the Black Chernozem (Mosier et al., 1998). Inconsistencies were also evident when comparing SS vs. PSS. There were no significant differences in N_2O fluxes between the two digested manure forms except when applied at the 400 mg N kg^{-1} rate to the Black Chernozem, in which case the fluxes were significantly higher for PSS than SS at all but the first sampling date. The unexpectedly higher emissions from PSS- compared to SS-amended soils in the Black Chernozem were likely due to greater heterogeneous dispersion of active denitrification microsites in the PSS-amended soil (Parkin, 1987) from the concentrated denitrifiable NO_3^- (high concentration of N in pellets) and organic C (substrate for microbes). Parkin (1987) found denitrification to be encouraged by a combination of anaerobic conditions, glucose and nitrate, all of which were likely greater in the Black than the Dark Brown Chernozem, reflecting the importance of soil by amendment interaction effects on N forms and dynamics.

Laboratory analysis indicated that soluble inorganic N after pelletization was only 9% of that in the SS prior to pelletization (Table 2.2), probably due to loss of dissolved N during pelletization. The difference in N_2O emissions between SS and PSS in the Dark Brown Chernozem is particularly surprising considering that SS had the highest moisture content, which was expected to create more anaerobic conditions and therefore greater N_2O emissions than raw manure and PSS. However, at 70% FC moisture content, it is likely that the Dark Brown Chernozem, with its conceivably large pore volume, was well aerated so that addition of the SS did not disturb the soil air/water balance sufficiently to induce denitrifying conditions (Linn and Doran, 1984). The greater emissions from manure than SS were likely due to the greater NO_3-N concentration in manure-amended soils from the higher NH_4-N available for nitrification in the manure treatments (Parkin, 1987). Conversely, the large emissions from PSS-amended soils may be due to the greater concentration of N in the pellets, creating hot spots for nitrification and denitrification. By comparison, SS was homogenously mixed with soil and did not have high N concentration spots.

The Black Chernozem had greater N_2O emissions than the Dark Brown Chernozem, possibly due to greater inorganic N and organic C contents of the Black Chernozem since N_2O is mostly produced during nitrification and denitrification, processes that are driven by microorganisms and depend on factors such as inorganic N content, organic C, and aeration (Bouwman et al., 2002; Velthof et al., 2003). The Black Chernozem's greater clay and organic matter contents probably led to a higher FC so that at 70% FC, the soil had more water and less air space relative to the Dark Brown soil, favouring the formation of anaerobic microsites that promoted denitrification (Brady and Weil, 2008). Therefore, even with the presumably good aeration at the 70% FC soil moisture content used, greater denitrification in the Black Chernozem (higher NO_3^- and C) than in the Dark Brown Chernozem was probably due to higher O_2 consumption rates in the former (Parkin, 1987; Dalal et al., 2009). Soils with high organic C and inorganic N concentrations, like the Black Chernozem (91 g C kg^{-1} and 42 mg N kg^{-1}, respectively) are prone to high N_2O emissions, indicating the influence of soil fertility on N_2O emissions (Bouwman et al., 2002).

Nitrous oxide was likely produced from both nitrification and denitrification, perhaps initially from nitrification of NH_4-N contained in the amendments and subsequently from denitrification of NO_3-N accumulated in the soil due to mineralization of organic N in the amendments (Dalal et al., 2009). The delayed peak of N_2O flux in PSS-amended soils (highest on day 3) relative to the other amendments (highest on day 0) can be attributed to the lower initial NH_4-N content of PSS (Table 2.2). Nitrous oxide emission peaks within 48 hr of amendment application have been reported (Yang et al., 2002), indicating that nitrification proceeds shortly after amendment application. Although we did not measure the two processes individually to determine their relative contributions towards N_2O emission, it is probable that denitrification was more dominant, due in part to greater emissions from the organic amendments relative to UMP. Unlike the UMP treatment, application of manure, SS, and PSS increased microbial oxygen demand primarily due to greater C substrate supply and utilization (indicated by the greater CO_2 production), thus creating anaerobic conditions conducive to denitrification and N_2O emission (Dalal et al., 2009).

The increase in N_2O fluxes with increasing N rate observed in our study (Fig. 2.3b) is consistent with observations by others. For example, Velthof et al. (2003) reported a linear relationship between N application rate and N_2O fluxes during the first 3 wk following application of ammonium nitrate (NH_4NO_3) in a 98-d incubation experiment. In the present study, however, this effect varied with amendment type. The N rate effect is a direct result of higher inorganic N concentration in the soil prompting greater N_2O-N losses (Bouwman, 1996; Mosier et al., 1998; Bouwman et al., 2002). Using information from 846 N_2O emission measurements in agricultural fields, Bouwman et al. (2002) also reported an increase in fluxes with increasing N rate, indicating the importance of N application rate for sustainable nutrient management and GHG modeling and inventory.

Nitrous oxide fluxes from soils amended with UMP were lower than N_2O losses from manure- and ADM-amended soils. Exceptions to this were fluxes from the 800 mg N kg^{-1} UMP rate in the Dark Brown soil that were the highest from day 28 until the end of the incubation (Fig. 2.1b). Lower fluxes in UMP-amended soils can be explained by the lack of a C substrate in the fertilizer, which limits the growth of denitrifying microorganisms (Parkin, 1987). Applying manure, SS, and PSS to soil promoted conditions that favoured denitrification due to increased concentrations of easily decomposable organic C and inorganic N, and by forming anaerobic microsites due to enhanced microbial respiration (Flessa and Beese, 2000). Rochette et al. (2008) reported very low to barely detectable (on most sampling dates) N_2O emissions from plots receiving NH_4NO_3 fertilizer compared with significantly higher emissions in manured plots; they attributed the lower fluxes to low C availability in the fertilizer-amended soil, which reduced denitrification. On the contrary, Bouwman et al. (2002) reported that emissions were 20% lower from agricultural fields amended with livestock manure compared with those receiving inorganic N fertilizers. The discrepancy in relative quantities of N_2O fluxes measured from manure- vs. fertilizer-amended soils in laboratory and field experiments can be explained by greater NH_3 volatilization from manure relative to fertilizer-amended plots in the field, which reduces the quantity of manure N that can be lost as N_2O (Rochette et al., 2008).

At the lower N application rate, N_2O fluxes had levelled off by day 42 for the Dark Brown Chernozem and about one week later for the more fertile Black Chernozem. These periods are comparable with the 40 d period reported by Rochette et al. (2008) for field N_2O fluxes following fertilization. Therefore, the short duration of N_2O fluxes indicates that the enhanced N_2O emissions triggered by manure-, SS-, and PSS-derived C and N substrates are often short-lived (Rochette et al., 2008).

Total N losses as a percentage of total applied N were 1.4, 0.4, 2.3, and -0.01% for manure, SS, PSS, and UMP, respectively, at the 400 mg N kg^{-1} rate. These N_2O losses are comparable to those reported by Akiyama et al. (2004) from a 38-d incubation study of a sandy loam soil amended with poultry litter, composted plant residues, sewage sludge pellets, cattle farmyard manure, and urea at rates equivalent to 120 kg N ha^{-1}. In this study, 0.01-1.65% of N applied with organic amendments and 0.04-0.62% of urea-N were denitrified. Results from the present study suggest the suitability of the Intergovernmental Panel on Climate Change (IPCC) N_2O emission factor of 1% (IPCC, 2006) for use on raw feedlot manure-, SS-, and UMP-amended soils. The PSS-treated soil at both rates, and the raw manure-amended soil at the 800 mg N kg^{-1} rate, emitted higher N_2O than suggested by the IPCC guideline for manure and its derivatives.

2.5.2 Carbon dioxide emissions

During the initial stages of organic material decomposition in soil, there is a rapid increase in the population of heterotrophic microorganisms, indicated by increased evolution of CO_2 (Havlin et al., 2005). As mentioned above, the highest fluxes of CO_2 were measured from manure-amended soils immediately after incubation setup. The high fluxes from manure application are due to the release of CO_2 dissolved in manure and CO_2 formation from HCO_3^- and CO_3^{2-} species dissolved in manure (Flessa and Beese, 2000).

The higher cumulative CO_2 losses in soils amended with SS compared with those amended with raw manure and PSS were likely due to higher microbial activity in SS-amended soils resulting from enhanced soil moisture content following SS application. Aerobic microbial activity, hence CO_2 evolution, increases with soil water content (Linn and Doran, 1984). The lowest cumulative CO_2 emissions (hence lowest microbial activity) were measured in UMP- amended soils. This is expected because microbes need organic C for growth and energy so that application of UMP, which supplies only N, stimulated smaller microbial growth than the organic amendments which supplied both N and C (Flessa and Beese, 2000; Rochette et al., 2008).

Carbon dioxide fluxes in non-amended soils were highest soon after set-up and decreased to almost constant levels after the first week. By contrast, Flessa and Beese (2000) reported constant CO_2 fluxes from non-amended soils, without the initial linear decrease observed in the present study. This discrepancy may be due to the residual effects of re-wetting previously air-dry soil, suggesting that the 2-d pre-incubation period that we used prior to experiment setup was inadequate to suppress the initial CO_2 flux due to soil re-wetting (Bertora et al., 2008). In the study by Flessa and Beese (2000), soils were pre-conditioned for 4 weeks before amendments were applied. Nitrifying and denitrifying microbial organisms are well adapted to surviving dry conditions and to becoming active soon after soil re-wetting (Davidson, 1992).

Although the static incubation method used in the current study measures N_2O and CO_2 evolution fairly well (Rolston, 1981), it assumes a constant flux rate during the 2 h that gas is allowed to accumulate before a sample is taken (Dalal et al., 2003). Gas concentration gradient decreases with time due to the accumulation of diffusing gas in the jar or chamber, affecting the accuracy of gas concentration measurements (Rolston, 1981). Additionally, covering jars for 2 h may have affected soil temperature and gas composition (Rolston, 1981; Dalal et al., 2003). However, since the resulting error in gas fluxes is due to the method used, all experimental units were presumably affected in the same way so that relative differences among treatments were not altered.

2.5.3 Net soil inorganic nitrogen content after incubation

During separation of SS from the liquid fraction, most of the readily available N is removed in the liquid fraction, leaving less-readily degradable organic N in SS. This explains the lower net mineralization measured in SS- than in non-amended soils. Pelletization further lowers the available N content of ADM through loss of dissolved N during pelletization, hence the higher C/N ratio of PSS and greater N immobilization in PSS-amended soils. This immobilization was more pronounced in the Black Chernozem than in the Dark Brown Chernozem, ostensibly due to greater microbial populations in the more fertile Black Chernozem with greater initial inorganic N and organic C contents. The higher N immobilization in PSS-amended soils at the 800 than at the 400 mg N kg^{-1} rate was due to greater amounts of PSS prompting greater microbial demand for inorganic N to meet microbial N requirements for protein synthesis and the concomitant decomposition of organic material (Havlin et al., 2005), causing a depletion of soil solution N. The high residual inorganic N contents and low CO_2 fluxes in soils amended with UMP vs. those receiving organic amendments (SS, PSS, and manure) indicate the readily available nature of N in UMP vs. the organic amendments.

2.6 Conclusions

Amendment effects on N_2O emissions varied with soil type, amendment rate, and sampling time. For both soils, cumulative N_2O emissions from manure- and PSS-amended soils were not significantly different. However, cumulative N_2O emissions from PSS- and SS-amended soils were significantly different in the Black Chernozem but not in the Dark Brown Chernozem. Increasing the N rate from 400 to 800 mg N kg^{-1} caused a significant increase in cumulative N_2O emissions in soils receiving raw manure application but not in those amended with SS and PSS. Microbial activity, as indicated by CO_2 fluxes, was greatest in SS-amended soils but this enhanced microbial activity was not accompanied by high N_2O emissions. These findings have important implications for the use of ADM as a nutrient source for crop production. Although mineral N content was lower in SS than manure-amended soil at the end of the incubation, SS application resulted in lower N_2O emissions. On the other hand, application of PSS to soils was not beneficial, as shown by the high N_2O emissions and net N immobilization in PSS-amended soils. With the growing concern over N_2O emissions, farmers may opt to use SS in place of raw manure to reduce N_2O emission from their fields if similar effects of SS application can be demonstrated under field conditions.

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3. NITROGEN AND PHOSPHORUS MINERALIZATION CHARACTERISTICS OF ANAEROBICALLY DIGESTED FEEDLOT MANURE APPLIED TO CHERNOZEMIC SOILS

3.1 Abstract

Knowledge of nutrient release following application of organic amendments to soil is important for their sustainable use as nutrient sources for crops. Anaerobically digested manure (ADM) is a nutrient-rich co-product from manure-based biogas plants that is often separated into solid and liquid fractions that are applied to cropland at rates based on those for raw manure. Application of ADM could result in different nitrogen (N) and phosphorus (P) mineralization patterns compared to raw manure due to biophysicochemical changes occurring during anaerobic digestion. We tested this hypothesis using a 10-wk laboratory incubation experiment in which a Dark Brown Chernozem (Typic Haploboroll) with a clay loam texture and a Black Chernozem (Typic Haplocryoll) with a silty clay texture were amended with two N-based rates (400 and 800 mg N kg^{-1} soil) of raw beef cattle (*Bos taurus*) feedlot manure, the separated solids fraction of ADM (SS), pelletized SS (PSS), and urea + monoammonium phosphate (UMP). Across rates, net mineralized N concentrations in soils amended with UMP, manure, SS, and PSS were 45, 24, 0.25, and -16% of added N, respectively, at the end of the 10 wk. All treatments had positive net P mineralization throughout the 10 wk. The lower N mineralization potential of SS and PSS compared with manure suggests that digested manure may have to be either applied at higher rates compared with raw manure or supplemented with inorganic N fertilizer to ensure adequate N supply for plants.

However, use of higher SS and PSS rates could increase the potential for P accumulation in receiving soils, hence the risk of P loss to surface water systems.

3.2 Introduction

The application of organic amendments to agricultural land in an effort to reduce costs and environmental concerns associated with land-filling and incineration of organic wastes, and to reduce reliance on synthetic fertilizers is increasing (Chae and Tabatabai, 1986; Yang et al., 2004). Organic amendments improve soil physical properties and supply micro- and macronutrients essential for crop production (Eghball, 2002; Havlin et al., 2005). However, little is known about the nutrient release patterns of most organic amendments (Helgason et al., 2007; Yang et al., 2004; Zvomuya et al., 2006). An understanding of nutrient release patterns following application of organic amendments to soil is fundamental to their sustainable use as plant nutrient sources (Eghball et al., 2002; Van Kessel et al., 2000).

Anaerobically digested manure (ADM) is a nutrient-rich substrate which is co-produced during biogas generation from anaerobic digestion of livestock manure. The ADM is often separated into solid (SS) and liquid fractions that are applied to agricultural land as a source of nutrients for crops. In western Canada, ADM is applied at the same rates (100 kg N ha^{-1} for forage barley, *Hordeum vulgare* L.) as raw manure. The possibility of biophysicochemical changes occurring during anaerobic digestion of raw manure and resulting in digested manure with altered nutrient release patterns has not been investigated. This is consistent with results from a recent incubation study (Chiyoka et al., 2011), which showed lower N_2O emissions from soils amended with SS than those amended with raw beef cattle feedlot manure.

Net mineralization or nutrient release is the outcome of two concurrent and oppositely directed processes: gross N mineralization and gross N immobilization (Luxhøi et al., 2006). Mineralization-immobilization turnover (MIT) has two distinct phases: a period of N or P accumulation, the duration and magnitude of which depends on substrate, and a period of net release (Bosatta and Berendse, 1984). During the accumulation phase, the N or P in the decomposing material is not readily accessible to microbes, which then utilize inorganic N or P from the soil (net immobilization) for protein synthesis (Havlin et al., 2005). The period of microbial nutrient assimilation is followed by one of net nutrient release (net mineralization) following the death of microbial cells (Lockett, 1938 as cited by Singh and Jones, 1976).

Mineralization-immobilization turnover of N and P is influenced by both soil and amendment properties (Beauchamp and Paul, 1989). Results from controlled-environment experiments show that an amendment P content of at least 2 g kg^{-1} is required for P net mineralization to occur (Fuller et al., 1956) while amendment C:N ratios greater than 16-23 result in net N immobilization (Enwezor, 1976). Soil properties like clay content (Thomsen and Olesen, 2000) and soil environmental parameters such as moisture content (Agehara and Warncke, 2005) and temperature (Andersen and Jensen, 2001) influence MIT following the application of organic amendments to soil.

Several studies have quantified N and P mineralization following amendment application using incubation experiments (Enwezor, 1976; Loria et al., 2005; Burger and Venterea, 2008). Incubation studies eliminate environmental sources of variation, such as temperature and moisture content, which are important factors driving nutrient mineralization. These controlled-environment experiments are important for evaluating

N mineralization mechanisms (Eghball, 2002; Cabrera et al., 2005) and provide useful information for management and use of organic amendments.

The overall objective of this laboratory incubation experiment was to characterize the N and P release patterns of SS and pelletized SS (PSS) vs. raw beef cattle (*Bos taurus*) feedlot manure following application to two of the most common soils from central and southern Alberta, Canada.

3.3 Materials and Methods

3.3.1 Organic Amendments

Raw beef cattle feedlot manure (manure), SS, and pelletized SS (PSS) were collected into pails from a bio-gas plant in central Alberta, Canada. The amendments were stored at 4°C in sealed pails for 3 mo before incubation setup. The PSS consisted of cylindrical pellets that were approximately 9 mm long and 4 mm in diameter with a density of 1.2 Mg m^{-3}. Pelletization of livestock manure and other organic amendments is an increasingly common practice that reduces moisture content and increases economical hauling distance of manure, reduces odor, and improves ease of manure storage and handling (Hammac et al., 2007).

Amendment subsamples (10-g dry wt.) were freeze-dried, finely ground (<0.15 mm), and analyzed for total N and C (Table 3.1) by dry combustion (NS-2000 Nitrogen Analyzer, Leco Corporation, St. Joseph, MI). Inorganic N (NO_3-N, NO_2-N, and NH_4-N) concentrations (Table 3.1) were determined by the phenate method (Searle, 1984) on wet amendment samples (0.5 g) using a Model AA3 autoanalyzer (Bran+Luebbe, Nordersted, Germany) following extraction with 30 mL of 2 M KCl (Keeney and Nelson, 1982).

Total P was determined colorimetrically at a wavelength of 880 nm (Murphy and Riley, 1962) by a discrete autoanalyzer (Easy-Chem Pro, Sistea Scientific, LLC, Oak Brook, IL) following acid digestion with H_2SO_4 and H_2O_2 (Parkinson and Allen, 1975).

3.3.2 Soil Properties and Analysis

Two surface (0-15 cm) soils - a Dark Brown Chernozem (fine-loamy, mixed, Typic Haploboroll) and a Black Chernozem (silty clay Typic Haplocryoll) - were collected from Lethbridge (49°42'N; 112°47'W) and St. Albert (53°42'N; 113°47'W), respectively, in Alberta, Canada. The soils were air-dried, sieved through a 4-mm sieve and stored at room temperature (25±1.6°C) until the start of the experiment.

Sub-samples ground to pass through a 2-mm sieve were analyzed for particle size distribution (Gee and Bauder, 1986). Inorganic N and P concentrations were analyzed as described above for amendments following extraction of 5 g of soil with 25 mL of 2 M KCl and extraction of 1 g of soil with 20 mL of 0.5 M $NaHCO_3$ buffered at a pH of 8.5 (Olsen et al., 1954), respectively (Table 3.1).

Soil total C, N and P concentrations (Table 3.1) were determined as described for the organic amendments. Soil pH and EC were measured in 1:2 soils: water suspensions using Accumet AB and Accumet AB30 meters, respectively (Fisher Scientific, Hampton, NH). Soil moisture content at field capacity was determined under a suction of 30 kPa using a 500 kPa pressure plate moisture extractor (Model 1600, Soil Moisture Equipment Co., Santa Barbara, CA).

Table 3.1 Selected initial physical and chemical properties of soils and amendments used in the laboratory incubation study.

Property†	Dark Brown Chernozem	Black Chernozem	Raw Manure	SS‡	PSS§
Total N ($g\ kg^{-1}$)	2.2	7.1	18	15	15
Total C ($g\ kg^{-1}$)	22	80	355	450	438
C:N ratio	10	11	20	31	29
Total P ($g\ kg^{-1}$)	0.50	1.1	4.0	3.1	4.3
C:P ratio	42	78	89	145	102
NH_4^+-N (mg kg^{-1})	5	8	3493	2433	217
NO_3^--N ¶ (mg kg^{-1})	8	34	< 12	<12	<12
pH	6.3	6.3	7.8	8.7	7.6
EC (dS m^{-1})	0.17	0.24	11	5.0	7.0
Moisture content ($g\ kg^{-1}$)	-	-	628	745	91
Clay ($g\ kg^{-1}$)	294	451	-	-	-
Silt ($g\ kg^{-1}$)	271	480	-	-	-
Sand ($g\ kg^{-1}$)	435	70	-	-	-

† Moisture content and pH and electrical conductivity (EC) measured on fresh weight basis; total C, total N, NH_4^+-N, NO_3^--N, C:N ratio, total P and C:P ratio on a dry weight basis.

‡ Separated solids from anaerobically digested manure slurry.

§ Pelletized separated solids.

¶ NO_3^--N concentrations in manure, SS and PSS were below detection limit (<0.2 ppm). This threshold corresponds to <12 mg N kg^{-1} after factoring the dilution used for extraction, i.e., 0.5 g soil extracted in 30 mL of KCl.

3.3.3 Microcosm Setup and Sampling

Samples (100 g) of each soil were mixed with manure, SS, PSS, and a fertilizer blend (UMP) of urea (46-0-0) and monoammonium phosphate (11-52-0) at rates approximating 400 and 800 mg total N kg^{-1}, which approximate 100 and 200 kg N ha^{-1}, respectively. The lower rate (100 kg N ha^{-1}) is recommended for forage barley production in western Canada. A zero (non-amended) control treatment was included for comparison.

Because amendment rates were N-based, amounts of P applied varied among amendments. Thus, while P additions from manure and SS were similar (78 mg N kg^{-1} and 80 mg N kg^{-1}, respectively, at the 400 mg N kg^{-1} amendment rate), PSS supplied 128 mg P kg^{-1} soil at the same rate. For the UMP treatment, monoammonium phosphate was applied at 34 mg P kg^{-1} at both N rates.

Deionized water was added to non-amended and amended soils to bring the total soil moisture content to 70% of field capacity. The soils were packed into 120-mL plastic cups to bulk densities of 1.1 and 1.0 Mg m^{-3}, respectively, for the Dark Brown Chernozem and the Black Chernozem. Each cup was covered with a plastic cap that had four ~2-mm diameter holes to regulate moisture loss and gas exchange. All treatments were replicated three times in a split-split plot treatment layout with soil as main plot, amendment as subplot, and rate as sub-subplot. A total of 504 treatment units were prepared to accommodate seven sampling dates during the 10-wk incubation. The cups were placed in an incubator (Scientific IsoTemp Incubator Model 304, Pittsburgh, PA) set to 22°C. Plastic tubs (32 cm × 23 cm × 6 cm) were filled with water and placed in the incubator to reduce evaporation from the soils during incubation. Each cup (at 70% field capacity) was weighed soon after set-up and weekly thereafter to determine and replace, using deionized water, water lost by evaporation.

A set of 72 units apiece was taken out of the incubator at 0, 3, 7, 14, 28, 42 and 70 d, placed in sealed plastic bags, and stored frozen at -20°C until analysis. Soils were analyzed for pH, inorganic N, and inorganic P after determination of gravimetric moisture content. Net N and P mineralization amounts were calculated at each sampling time by subtracting the inorganic nutrient concentration in the non-amended soil from the inorganic nutrient concentration in the amended soil.

3.3.4 Statistical Analysis

Net mineralized N (N_{min}) and P (P_{min}) and pH (amended soils) data were analyzed using the MIXED procedure for repeated measures in SAS (Littell et al., 1998; SAS Institute Inc., 2008) with time as the repeated factor and using the spatial power [SP(POW)] covariance structure (Littell et al., 1998). Soil, amendment, and rate were fixed effects while replication and interaction of replication with the fixed effects were considered as random effects. Treatment differences were considered significant if $P < 0.05$ using the Tukey-Kramer method.

3.4 Results

3.4.1 Nitrogen Mineralization and Immobilization

Among the three organic amendments tested, net N mineralization was highest for manure (overall mean = 124 mg kg^{-1} soil) followed by SS (50 mg kg^{-1}), while PSS application resulted in net immobilization of N (net mineralization = -38 mg kg^{-1}) (Table 3.2). Differences among amendments, however, varied with sampling time and were affected by soil type and amendment rate, as shown by the significant ($P = 0.0001$) soil × amendment × rate × time interaction. Thus, while net N mineralization from manure was higher than that from PSS application throughout the sampling period, differences between manure and SS were generally not significant during the sampling period up to Day 7 but were significant at all sampling dates thereafter in both soils.

Inorganic N concentration of non-amended soils increased with time (Fig. 3.1). At both amendment rates, N_{min} was highest for UMP throughout the 70 d for both the Dark Brown and the Black Chernozem without the net N immobilization observed in the manure and ADM treatments (Fig. 3.1). In the UMP treatments at the lower rate, N_{min} consistently increased with time up to day 28 in the Dark Brown Chernozem and day 42 in the Black Chernozem. At the end of the 70-d incubation, only UMP and manure treatments had positive N_{min} in the Dark Brown Chernozem while all amendments except PSS had positive N_{min} in the Black Chernozem.

A rapid decline in N_{min} (N immobilization) occurred during the first week of incubation in soils amended with manure at both application rates (Fig. 3.1). A similar rapid N immobilization in SS-amended soils was observed only at the 400 mg N kg^{-1} rate in the Dark Brown Chernozem while net immobilization ($N_{min} < 0$) in PSS treatments was measured a week after incubation setup at both amendment rates in both soils.

Table 3.2 Soil, amendment, and rate effects on net mineralized nitrogen (Nmin) and phosphorus (Pmin) and pH of amended Dark Brown and Black Chernozemic soils during incubation.

Effect	Net Mineralized N	Net Mineralized P	Soil pH
	mg kg^{-1} soil		
Soil			
Dark Brown	84	25	6.4
Black	103	22	6.1
Amendment			
Manure	124	27	6.2
SS	50	42	6.4
PSS	-38	7.4	6.3
UMP	238	18	6.1
Rate (mg N kg^{-1})			
400	64	16	6.3
800	124	30	6.3
P-value			
Soil (S)	0.01	0.03	<0.0001
Amendment (A)	<0.0001	<0.0001	0.002
S × A	0.002	0.13	0.28
Rate (R)	<0.0001	<0.0001	0.37
S × R	<0.0001	0.53	0.57
A × R	<0.0001	<0.0001	0.05
S × A × R	<0.0001	0.72	0.04
Time (T)	<0.0001	<0.0001	<0.0001
S × T	0.05	0.02	<0.0001
A × T	<0.0001	<0.0001	<0.0001
S × A × T	<0.0001	0.23	<0.0001
R × T	0.40	0.54	<0.0001
S × R × T	0.04	0.38	0.71
A × R × T	<0.0001	0.64	0.23
S× A × R × T	0.0001	0.78	0.95

[†] Time main effects not presented because of the multiple sampling times. Some significant interaction effects which include Time as a factor are graphed.

Initial N immobilization in manure-amended soils was followed by N mineralization 2 wk after the start of incubation in the Black Chernozem and 2 wk later in the Dark Brown Chernozem. By comparison, net N immobilization continued until the end of the incubation in the Dark Brown Chernozem amended with SS and PSS and in the Black Chernozem amended with PSS. At the end of incubation, only UMP-amended soils had greater N_{min} than measured at the start of the experiment. Mineralized N concentrations in the Black Chernozem amended with manure at the 800 mg N kg^{-1} rate were similar for Days 0 and 70.

In the Dark Brown Chernozem, N_{min} values, averaged across soils, amendment rates, and sampling time, were significantly higher for SS than PSS during the 70-d incubation, except in samples taken on days 14, 28, and 42 at the 400 mg N kg^{-1} rate and on days 14 and 28 at the higher amendment rate when N_{min} was similar for the two amendments. In contrast, throughout the incubation and at both rates, SS-amended soils had higher N_{min} than PSS-amended soils in the Black Chernozem.

At the end of the incubation (Day 70), manure-amended soils had greater N_{min} than SS- and PSS-amended soils and at both rates. At the higher amendment rate, the difference in N_{min} between UMP and manure was greater in the Dark Brown (245 mg N kg^{-1}) than the Black Chernozem (132 mg N kg^{-1}) (Fig. 3.1). Across rates and sampling times, N_{min} was highest in UMP-amended soils (mean = 45% of applied N) and lowest in PSS-amended soils (-16% of applied N) while it was also higher with manure (24% of added N) than SS (1% of added N) application.

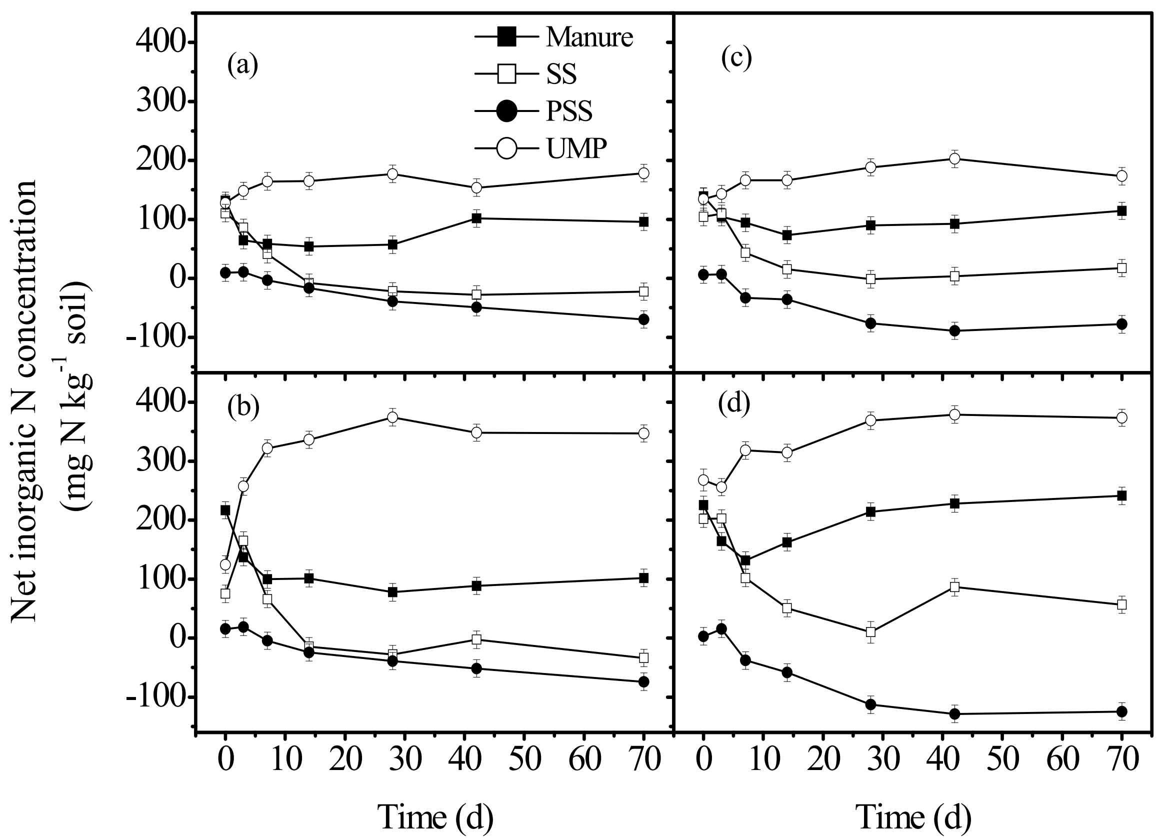

Figure 3.1 Net concentration of inorganic N in the Dark Brown Chernozem (a and b) and the Black Chernozem (c and d) amended with manure, separated solids (SS), pelletized SS (PSS), and monoammonium phosphate plus urea (UMP) at 400 (a and c) and 800 (b and d) mg N kg^{-1} soil rates. Dashed lines show mean inorganic N in non-amended soils.

3.4.2 Soil pH

Amendment effects on soil pH were significant (P = 0.002) but varied with soil, rate and sampling time (Table 3.2). Immediately after application, soil pH increased with application of SS and UMP but varied little with manure and PSS (Fig. 3.2). Overall, pH decreased with time and both soils had similar pH trends over the 70 d. However, pH was generally higher in the Dark Brown than the Black Chernozem.

In the Dark Brown Chernozem, SS- and PSS-amended soils had similar pH values at all sampling times except Days 0 and 3 when pH was significantly higher in SS-amended soils (Fig. 3.2a). Soil pH (i) decreased rapidly during the first week for all amendments; (ii) increased as of Day 14 and remained fairly constant until day 70 for PSS and SS treatments; (iii) continued declining up to Day 14 beyond which it remained constant until the end for the UMP-amended soil; and (iv) remained somewhat constant from Day 7 through Day 70 in manure-amended soils. At the end of the incubation, SS- and PSS-amended soils had similar and significantly higher pH values than manure- and UMP-amended soils.

There was no significant amendment effect on pH in the Black Chernozem throughout the 70-d incubation (Fig 3.2b). Soil pH of the amended Black Chernozem also declined during the first week but less rapidly than in the Dark Brown soil. In the Dark Brown Chernozem, soil pH was highest for SS and PSS at the 400 mg N kg^{-1} amendment rate and for SS at the 800 mg N kg^{-1} rate (Fig. 3.3a). The pH also increased with increasing manure and SS rates, decreased with increasing PSS rate, and did not vary with UMP rate. By comparison, pH in the Black Chernozem was similar and higher for SS and PSS than for manure and UMP at both rates (Fig. 3.3b).

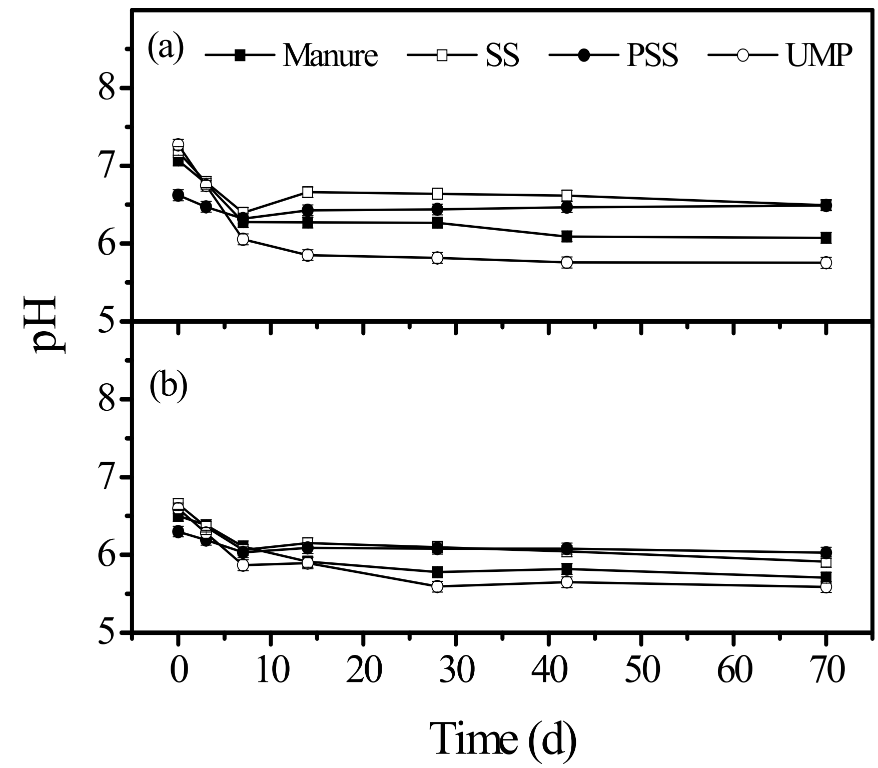

Figure 3.2 Change in pH of (a) Dark Brown and (a) Black Chernozemic soils amended with raw feedlot manure, separated solid fraction of anaerobically digested beef cattle feedlot manure (SS), pelletized SS (PSS), and monoammonium phosphate + urea (UMP), and incubated for 10-wk. Dashed lines show mean pH of control (non-amended) soils.

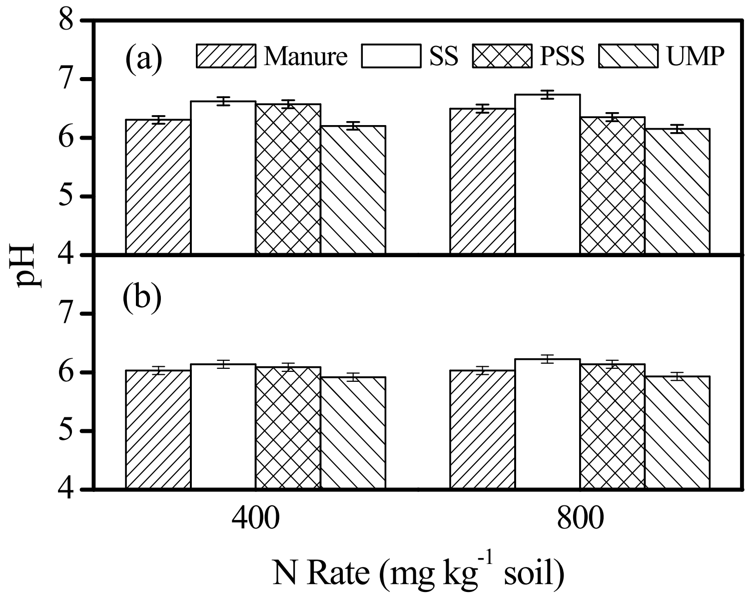

Figure 3.3 Soil pH of (a) Dark Brown and (a) Black Chernozemic soils amended with raw feedlot manure, separated solids (SS), pelletized SS (PSS), and a synthetic fertilizer blend of monoammonium phosphate and urea (UMP) at 400 and 800 mg N kg^{-1} soil rates, and incubated for 70 d.

3.4.3 Mineralized Phosphorus Concentration

Amendment × time ($p < 0.0001$), amendment × rate ($P < 0.0001$), and soil × time ($P = 0.02$) interactions were significant for P_{min} concentration (Table 3.2). All amended soils had greater P_{min} than non-amended soils throughout the 70-d incubation (Fig. 3.4). However, different extents of P immobilization were observed for the four amendments within the first 4 wk of incubation. Separated solids-amended soils had the highest P_{min} throughout the incubation period. Mineralized P peaked on Day 3 for SS but declined thereafter till Day 14, after which it remained fairly constant for the rest of the incubation. Mineralized P was lowest for PSS at all but the last sampling day when it was similar for PSS and UMP. On the last day of sampling, all amendments except SS had higher net P concentrations compared with the first sampling day.

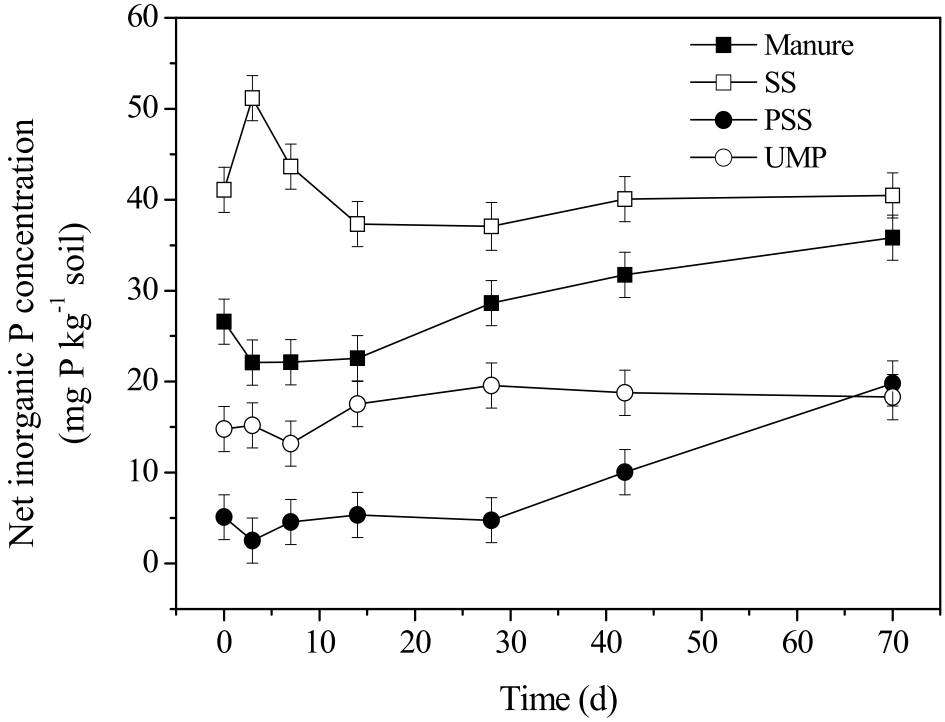

Figure 3.4 Net P concentration in Dark Brown and Black Chernozemic soils amended with raw feedlot manure, separated solid fraction of anaerobically digested feedlot manure (SS), pelletized SS (PSS), and monoammonium phosphate plus urea (UMP), and incubated for 10 wk. Dashed lines show mean inorganic N in non-amended soils.

Phosphorus mineralization, averaged across rates and amendments, was similar for the two soils, except on Days 28 and 70 when it was higher in the Dark Brown Chernozem (Fig. 3.5). On average, for the non-amended soils, the Black Chernozem had higher P_{min} than the Dark Brown Chernozem throughout the incubation period (Fig. 3.5). Net P_{min} was significantly higher at the higher than the lower N rate for all amendments except UMP (Fig. 6). Increasing SS rate resulted in the highest increase in P_{min} (30 mg P kg^{-1}) while increasing manure and PSS rates caused lower mineralized P increases of 18 and 6 mg P kg^{-1}, respectively.

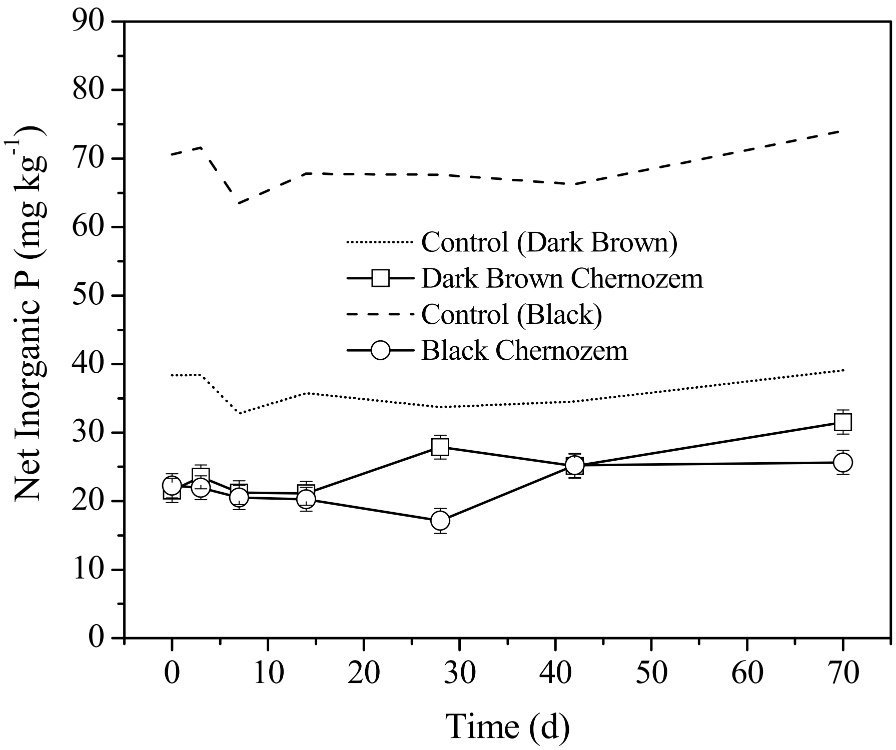

Figure 3.5 Net P concentration in Chernozemic soils amended with raw feedlot manure, separated solid fraction of anaerobically digested feedlot manure (SS), pelletized SS (PSS), and monoammonium phosphate plus urea (UMP), and incubated for 10 wk. Dotted and dashed lines show mean inorganic N in non-amended Dark Brown and Black Chernozemic soils, respectively.

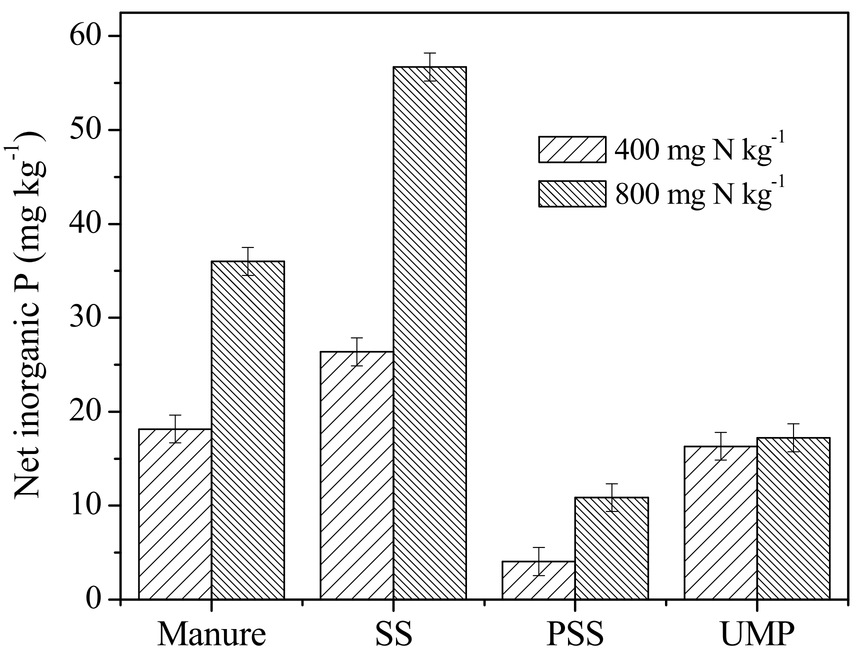

Figure 3.6 Effect of application rate (400 and 800 mg N kg^{-1}) of raw beef cattle feedlot manure, separated solids (SS), pelletized SS (PSS), and urea plus monoammonium phosphate (UMP) on net P concentration in Dark Brown and Black Chernozemic soils incubated for 10 wk.

3.5 Discussion

3.5.1 Mineralization and Immobilization of Nitrogen

The rapid N immobilization ($N_{min} < 0$) in manure- and SS-amended soils within 2 wk of amendment application was likely due to the presence of readily-decomposable organic C compounds (Flowers and Arnold, 1983; Zaman et al., 1998) in these amendments. Mineralization of N from soil-applied organic amendments occurs through the activity of heterotrophic microorganisms that require a readily available C substrate (Havlin et al., 2005). If the decomposing amendment is low in N concentration, microorganisms will utilize (hence immobilize) inorganic N (NO_3^- and NH_4^+) in the soil solution (Enwezor, 1976; Havlin et al., 2005), causing a decline in soil inorganic N. Burger and Venterea (2008) reported a 24- to 27-mg N kg^{-1} decrease in inorganic N concentrations of loam and clay loam soils amended with liquid dairy cattle manure (154 mg N kg^{-1} soil) during the first week of 180-d incubations at 10 and 25°C. Nitrogen immobilization was also observed during the early stages of an experiment in which a clay loam and a sandy loam amended with pig slurry (100 µg NH_4-N g^{-1} soil) were incubated at temperatures ranging from 5 to 30°C (Flowers and Arnold, 1983).

The high initial microbial N demand observed in the manure and SS treated soils in our experiment was likely due to labile C sources, such as volatile fatty acids (VFAs), which have been measured in manure slurries (Paul and Beauchamp, 1989; Kirchmann and Lundvall, 1993). Storage of manure and SS in sealed containers at 4°C prior to incubation set-up may have caused anaerobic conditions favoring the formation of VFAs (Burger and Venterea, 2008) that became a readily available C source for soil microorganisms. Paul and Beauchamp (1989) reported a decrease in VFA concentration in a silt loam amended with dairy cattle slurry, from 1200 to < 10 mg C kg^{-1} during the first 4 d of a 16-d incubation, and suggested immobilization rather than denitrification as the cause of nitrate disappearance during the rapid oxidation of VFAs. Kirchmann and Lundvall (1993) also observed sharp decreases in fatty acid concentrations in cattle- and pig slurry-amended silt loam within 2 d of incubation at 25°C. The amount of immobilized N has been found to be highly correlated (R^2 = 97%) with total fatty acid (C_2-C_6) content (Kirchmann and Lundvall, 1993). Microbes require enough N to give a C:N ratio of ~8:1 in their cells (Havlin et al., 2005), so that application of amendments with high C:N ratios (>25 – 30) (Shaffer and Ma, 2001; Stevenson and Cole, 1999) prompts the rapidly growing microbial population to utilize, hence immobilize, the inorganic N in the soil.

As amendment decomposition proceeds, the C:N ratio decreases due to the declining C concentration (evolved as CO_2 via respiration with small amounts converted to microbial biomass and synthesis products) and corresponding increase in N concentration, ultimately resulting in a new mineralization-immobilization equilibrium that favors net N mineralization (Havlin et al., 2005). In the present study, inorganic N immobilization was followed by N mineralization (an increase in N_{min}) after 7 to 28 d for manure and SS treatments depending on soil type (Fig. 3.1). Manure application resulted in higher N_{min} concentrations than SS and PSS, and this was related to the higher inorganic N concentration of manure (Table 3.1). Net N mineralization observed after 70 d for all amendments except SS and PSS in the Dark Brown Chernozem (Fig. 3.1a and b) and for all amendments but PSS in the Black Chernozem (Fig. 3.1c and d) indicates the influence of soil properties on MIT following amendment application.

The lower initial NH_4-N concentration and non-easily decomposable organic N of SS compared to raw manure explains the higher N immobilization in SS- compared with manure-amended soils. However, at the end of the 70-d incubation, SS application resulted in net immobilization in the Dark Brown Chernozem and net mineralization in the Black Chernozem. The higher initial inorganic N concentration and organic matter content of the Black Chernozem likely reduced N immobilization compared to the Dark Brown Chernozem, thereby masking the greater immobilization that would have otherwise occurred. Lower inorganic N concentration and organic matter content in the Dark Brown Chernozem stimulated greater microbial N immobilization relative to N mineralization (hence net N immobilization). The Black Chernozem with higher clay content (450 g kg^{-1} soil) than the Dark Brown Chernozem (300 g kg^{-1} soil) likely had a higher rate of soil N MIT, which reduced immobilization in the Black Chernozem vs. the Dark Brown Chernozem when amended with SS, an amendment with intermediate inorganic N concentration. This is consistent with Thomsen and Olesen (2000) who reported an increase in inorganic N concentration with increasing clay content of non-amended soils incubated for 266 d, from 48 mg N kg^{-1} in the soil with the lowest clay content (110 g kg^{-1} soil) to 84 mg N kg^{-1} in the soil with the highest clay content (480 g kg^{-1} soil).

The apparent lack of subsequent net N mineralization of amendment organic N or previously immobilized N observed in the SS- and PSS-amended Dark Brown Chernozem and the PSS-amended Black Chernozem after 70 d of incubation has been reported in other extended incubation studies. For example, at the end of a 180-d incubation, Burger and Venterea (2008) observed lower inorganic N concentrations in liquid dairy cattle manure-amended soils than in non-amended soils. Similarly, Flowers and Arnold (1983) observed N immobilization in pig manure slurry-amended soil after incubation for 175 d. Gagnon and Simard (1999) also reported high immobilization following a 13-wk incubation of a sandy loam soil amended with fresh solid beef cattle manure and young dairy cattle manure compost. These findings indicate that MIT is slow and suggest that plants may not derive any benefit from the immobilized N during a normal growing season (Beauchamp and Paul, 1989). Results from the present study suggest that it may be prudent to supplement field applications of SS and PSS with inorganic N fertilizer to ensure earlier release of N and improve soil productivity (Ndayegamiye and Isfan, 1991).

The positive net N_{min} observed throughout the incubation in soils amended with UMP was likely due to the fertilizer's readily available inorganic N and smaller C substrate (compared to the organic amendments) needed to stimulate microbial growth and N immobilization. Although N_{min} did not decline in the UMP-amended soils, especially immediately following incubation setup, N immobilization probably occurred concurrently with, but at a lower rate than N mineralization (Burger and Venterea, 2008). This partly explains the relatively low net mineralized N concentration in UMP treatments (45% of added N) measured on day 70. The steady accumulation of N_{min} in UMP-amended soils after the initial mineralization flush was likely due to mineralization of soil organic N and/or remineralization of previously immobilized N.

Despite the increase in soluble inorganic N associated with anaerobic digestion of livestock manure (Bernal and Kirchmann, 1992; Kirchmann and Lundvall, 1993; Loria and Sawyer, 2005), solid/liquid separation of the digestate (ADM) partitions most of the available N into the liquid fraction, leaving the SS with lower available N concentrations than the raw manure (Table 1). Also, ADM is characterized by the presence of energy-rich and easily decomposable C, which is conducive to N immobilization (Bernal and Kirchmann, 1992). This explains the greater immobilization observed in SS than manure treatments. Pelletization further lowers the available N content of ADM through loss of dissolved N during pelletization, hence the higher N immobilization in PSS- than in SS-amended soils. Differences in inorganic N concentrations of the amended soils at the end of the incubation reflect the effect of initial amendment NH_4-N concentration on N mineralization (Beauchamp and Paul, 1989). Soils amended with manure (initial NH_4-N concentration ~3500 mg N kg^{-1}) had greater N_{min} than SS-amended soils (initial NH_4-N concentration ~2400 mg N kg^{-1}), which in turn had higher N_{min} than PSS-amended soils (initial NH_4-N ~ 220 mg N kg^{-1}).

Whether N is mineralized or immobilized depends on the C:N ratio of the organic material being decomposed by microorganisms (Havlin et al., 2005). Studies indicate that the critical C:N ratio above which net mineralization of amendments is unlikely ranges from as low as 16 to >20 (Beauchamp and Paul, 1989; Calderon et al., 2005; Enwezor, 1976; Havlin et al., 2005) to as high as 25 to 30 (Shaffer and Ma, 2001; Stevenson and Cole, 1999). Results from the present study are in partial agreement with these reported C:N ratio thresholds. Raw manure (C:N ratio = 20) had net mineralization at the end of the incubation. However, contrary to C:N ratio predictions, application of SS (C:N ratio =

31) resulted in net N mineralization, but only in the Black Chernozem, indicating that mineralization of N following amendment application is highly dependent on both amendment type and soil organic matter content (Beauchamp and Paul, 1989).

For the non-amended soils, inorganic N concentrations throughout the incubation period were higher in the Black than the Dark Brown Chernozem. This is likely due to the different initial N and C economies of these soils. The Black Chernozem had higher initial inorganic N (42 mg N kg^{-1}) and organic C (91 g C kg^{-1}) concentrations than the Dark Brown Chernozem (13 mg N kg^{-1} and 31 g C kg^{-1}, respectively). These findings are consistent with results reported by Chae and Tabatabai (1986), who found greater N mineralization in a non-amended fine montmorillonitic soil (inorganic N = 16 mg N kg^{-1} and organic C = 58 g kg^{-1}) than a non-amended fine loamy soil (inorganic N = 7 mg N kg^{-1} and organic C = 13 g kg^{-1}). For manure and SS treatments, immobilization was greater in the Dark Brown Chernozem than in the Black Chernozem, likely due to the greater inorganic N which reduced immobilization in the Black soil.

Higher amendment rate was associated with greater N_{min} in UMP-amended soils (both soils) and in the manure-amended Black Chernozem likely due to the higher inorganic N concentration at the higher rate (800 mg N kg^{-1}) with no corresponding increase in microbial demand for N. The greater N immobilization at the higher amendment rate observed for all three organic amendments in the Dark Brown Chernozem and for SS and PSS in the Black Chernozem was probably due to higher C substrate-stimulated microbial growth and hence N demand for microbial protein synthesis (Enwezor, 1976; Havlin et al., 2005). These findings are consistent with those of Flowers and Arnold (1983), who observed greater N immobilization in soils amended with pig slurry applied at 250 than at 100 μg N g^{-1} soil.

To be able to determine the potential N release pattern of an organic amendment following application to soil, gaseous losses need to be estimated parallel to the determination of inorganic N changes during incubation (Kirchmann and Lundvall, 1993). Ammonia volatilization and denitrification were assumed negligible in the present study since amendments were mixed with soil and microcosms were covered. However, the low inorganic N concentrations for all treatments suggest that one or both of these alternative N pathways (e.g., N volatilization from manure and SS during application and soil mixing, and denitrification during incubation) may have been significant. Ammonia volatilization was more likely for SS treatments that had higher soil pH (6.4) than the other treatments. According to Mkhabela et al. (2006), NH_3 volatilization N losses are high at soil pH ≥ 6.3. As such, reliance on soil inorganic N changes as the sole indicator of potential N mineralization may have overestimated the amount of N immobilized because some inorganic N may have been volatilized or denitrified (Calderon et al., 2005; Kirchmann and Lundvall, 1993). However, results from an incubation study that examined N_2O emissions (Chiyoka et al., 2011) showed low N_2O-N losses of 1.4, 0.4, 2.3, and -0.01% of total N applied with manure, SS, PSS, and UMP, respectively, at the lower rate. By comparison, N_2O losses greater than 10% of total manure-applied N have been reported from other incubation studies (Velthof et al., 2003).

Storage of the organic amendments at 4°C for 3 mo before incubation set-up likely did not fully suppress microbial activity and probably allowed decomposition of raw manure and SS during storage, resulting in the high (> 100 mg N kg^{-1}) N_{min}

concentrations on Day 0. Although incubation studies are important for evaluating N mineralization mechanisms, relating the incubation results to field conditions can be difficult (Eghball, 2000; Eghball et al., 2002), partly due to soil disturbance and use of an artificially created environment (Rees, 1989). Nonetheless, such microcosm studies are essential for the characterization of mechanisms controlling nutrient dynamics and usually offer useful insights into processes occurring under field conditions.

3.5.2 Soil pH

The initially higher pH of soils amended with SS and manure relative to the control, PSS, and UMP treatments was likely due to manure alkalinity and carbonate concentrations (Chantigny et al., 2004). The pH was also higher in SS- than manure-amended soils, which is consistent with the higher pH (8.7) of SS compared with manure (7.8) (Table 3.1). High pH probably increased NH_3(aq) concentration, hence the potential for surface NH_3 volatilization (Loria and Sawyer, 2005) in SS, leading to the observed lower N_{min} than manure.

The rapid decline in soil pH soon after the start of incubation was likely due to nitrification, a proton (H^+)-generating reaction which is, therefore, acidifying (Bernal and Kirchmann, 1992). Lack of preconditioning of the air-dried soils prior to amendment application and watering to 70% field capacity probably caused a flush in microbial activity, hence the rapid decrease in pH (even in non-amended soils) since nitrifying microorganisms have excellent survivability in dry conditions and become active soon after soil rewetting (Davidson, 1992). This rewetting effect was also evident in N_{min} dynamics, especially in manure- and SS-amended soils (Fig. 3.1).

The increase in soil pH in all treatments measured 2 wk after the start of incubation may be indicative of the end of rewetting effects. Also, this peak may be due to aerobic decomposition of the amendments (a process that has an alkalinizing effect) (Bernal and Kirchmann, 1992) having a greater magnitude than nitrification, an acidifying process. Thereafter, nitrification of mineralized NH_4-N caused a steady decline in pH.

3.5.3 Mineralization and Immobilization of P

All amendments resulted in net P mineralization ($P_{min} > 0$) throughout the incubation period. The threshold P concentration of organic amendments above which there is no net immobilization of P by soil microorganisms has been shown to range between 2 g kg^{-1} (Fuller et al., 1956) and 3 g kg^{-1} (Singh and Jones, 1976), a value exceeded by the total P concentrations of manure, SS, and PSS in the present study (Table 3.1). Amendment C:P ratio determines the predominance of P mineralization over immobilization (Enwezor, 1976; Havlin et al., 2005; Laboski and Lamb, 2003) and a C:P ratio < 200 results in net mineralization of organic P (Havlin et al., 2005), as was observed in the present study.

Inorganic P concentrations of non-amended soils (control) remained fairly constant during the incubation (50-55 mg P kg^{-1}). Duffera et al. (1999) reported similar findings from a 16-wk incubation of loamy sand, clay loam, sandy loam, and coarse loamy soils amended with processed swine lagoon solids. Application of amendments likely disturbed the soil P balance causing a decline in P_{min} concentrations during the first 2 wk of our incubation experiment, after which P_{min} remained constant (SS and UMP) or increased (manure and PSS). The initial decline was likely due to P immobilization by microorganisms and/or P adsorption by soil mineral and clay surfaces (Fuller et al., 1956;

Havlin et al., 2005). However, this decline was small and did not cause net P immobilization.

The continued increase in P_{min} in manure and PSS treatments (Day 28 onwards, Fig. 3.4) was likely due to the decomposition of these amendments, which resulted in concentrations of organic acids that effectively reduced P sorption to the soil and increased P availability (Fuller et al., 1956; Havlin et al., 2005; Laboski and Lamb, 2003). In contrast, P_{min} in UMP-amended soils declined consistently from Day 28 until the end of incubation (Fig. 3.4), which could be partly due to P adsorption by soil mineral and clay surfaces (Fuller et al., 1956). A low C:P ratio (hence limited C substrate) in the UMP treatment restricted microbial growth, thereby limiting P immobilization.

Although P_{min} concentrations of manure and SS varied during the incubation, at the end of the 70 d incubation, the two sources had similar P_{min}. While N mineralization is governed primarily by microbial processes, availability of P is influenced by both biological and chemical processes (i.e., microbial driven mineralization and adsorption) (Havlin et al., 2005). However, to a greater extent, the N and P concentration of any organic amendment in relation to microbial demand for these elements governs the N and P release pattern of the amendment (Enwezor, 1976). Over time, manure- and ADM-P underwent both mineralization and adsorption whereas orthophosphate-P in the inorganic fertilizer (UMP) was largely affected by adsorption (Loria and Sawyer, 2005).

As the rate of manure, SS, and PSS application increased, P_{min} also increased (Fig. 3.6). This is consistent with studies by Duffera et al. (1999), Laboski and Lamb (2003), and Loria and Sawyer (2005), which showed an increase in plant-available P with increasing manure application rate. It is noteworthy that although P availability increased with increasing manure, SS, and PSS rate, such an increase was not observed in N_{min} of these amendments. In fact, N immobilization increased with increasing manure, SS, and PSS rate so that N_{min} at the 800 mg N kg^{-1} rate was lower or slightly higher than that at 400 mg N kg^{-1} (Fig. 3.1).

Because the amendments used in the present study supplied different amounts of P when applied to supply 400 and 800 mg N kg^{-1}, the different initial concentrations of P could have influenced P_{min} patterns and magnitude. However, this is unlikely because when the P_{min} data were analyzed as percentage changes in P with time for each treatment or with initial P concentration as a covariate, there were no significant treatment effects (data not presented).

Results from the present study indicate the potential for P accumulation, especially if organic amendments are added to meet crop N requirements (Eghball and Power, 1999), more so because these amendments (at the lower rate) supplied at least double the amount of total P normally added as inorganic fertilizer. Additionally, the N:P ratios of manure, SS and PSS (3.5:1 to 4.7:1) are narrower than the N:P uptake ratios of most crops (e.g., 6:1 for corn) (Eghball et al., 2005), resulting in soil P accumulation and increased potential for P environmental pollution.

3.6 Conclusions

Anaerobically digested feedlot cattle manure has different nutrient release patterns from raw (non-digested) feedlot cattle manure. Application of SS resulted in lower soil available N compared with raw manure. Although SS does provide plant available N, the rate of N mineralization may not be rapid enough to meet plant N requirements during rapid growth. Nitrogen immobilization in SS treatments increased with decreasing initial soil total N concentration and organic matter content. Pelletizing SS, though favorable for ease of haulage, storage, and handling, results in a less degradable amendment (PSS) which is more prone to N immobilization following soil application, potentially causing N deficiency and low crop yields. Mineralization patterns of manure, SS, and PSS reflected the amendments' initial inorganic N concentrations.

Unlike the net N immobilization observed for manure, SS, and PSS, net P immobilization was not observed during the incubation. Increasing manure, SS, and PSS rates resulted in higher P mineralization in contrast to mineralized N, which decreased with increasing rate of these amendments (except for manure in the Black Chernozem). The combination of higher P availability and greater N immobilization at higher amendment rates is a cause for concern because of the increased potential for P accumulation and environmental pollution.

At the end of the 70-d incubation, manure and SS treatments had similar net mineralized P concentrations, suggesting that if applied at equivalent total N rates, raw manure and SS will supply similar plant-available P. However, because of lower N availability in SS- than manure-amended soils, supplemental inorganic fertilizer N may have to be applied with SS (and PSS) to increase the N:P ratio and increase the plant-available N early in the growing season.

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4. NITROGEN AND PHOSPHORUS UPTAKE BY BARLEY GROWN IN CHERNOZEMIC SOILS AMENDED WITH ANAEROBICALLY DIGESTED FEEDLOT MANURE

4.1. Abstract

Anaerobically digested feedlot manure (ADM) is the nutrient-rich by-product of biogas production from anaerobic digestion of livestock manure. It is often separated into solid and liquid fractions that are applied to cropland as nutrient sources for crops based on rates for raw (non-digested) manure. However, physical, chemical and biological changes during the digestion process may result in ADM with different N and P supply patterns, hence requiring different application rates, compared with raw manure. A growth room experiment comprising five, 6-wk long growth cycles of forage barley (*Hordeum vulgare* L.) was conducted to test this hypothesis. Forage barley plants were grown in pots containing 1.5 kg apiece of a Dark Brown Chernozem (Typic Haploboroll, clay loam) and a Black Chernozem (Typic Haplocryoll, silty clay) that were amended with raw beef cattle (*Bos taurus*) feedlot manure, separated solids fraction of ADM (SS), pelletized SS (PSS), and urea + monoammonium (UMP) at two nitrogen- (N-) based rates (400 and 800 mg N kg^{-1} soil). A zero amendment (control) treatment was included for comparison. All treatments initially received a nutrient solution which excluded N and phosphorus (P) to ensure an adequate supply of all other essential nutrients. Biomass yield was similar for manure, SS, and UMP (mean = 32 g kg^{-1} soil) and lowest for PSS (18 g kg^{-1} soil). Net N uptake was highest for manure (232 mg N kg^{-1}) followed by SS

(140 mg N kg^{-1}) while plants grown in PSS-amended soils in fact took up 61 mg N kg^{-1} less than those in the control (i.e., net N uptake = -61 mg N kg^{-1}). Net P uptake was also similar (mean = 26 mg P kg^{-1}) for manure and SS and significantly lower for PSS (10 mg P kg^{-1}). Net uptake of both N and P increased with increasing rate of manure, SS, and UMP but decreased with increasing PSS rate. This suggests that pelletization of SS results in the loss of labile and readily available N and C compounds leaving recalcitrant constituents that are not easily degradable by soil microbes. Although biomass yields were similar for raw manure and SS, slower organic N mineralization and lower apparent N recovery in SS-amended soils may limit the nutrient supply capacity of SS. It may be prudent to apply SS and PSS at higher rates than manure or to supplement these digested manure forms with mineral N fertilizer to ensure adequate available N for plant uptake.

4.2. Introduction

Anaerobically digested manure (ADM) is a nutrient rich substrate that is co-generated during biogas production from anaerobic digestion of livestock manure. The ADM slurry is often separated into solid and liquid fractions that are applied to cropland as a source of nutrients for crops. In western Canada, beef cattle manure is often applied to forage barley (*Hordeum vulgare* L.) at rates equivalent to 100 kg N ha^{-1} (Alberta Agriculture and Rural Development, 2008). The same rate has been adopted for ADM application (Hao, personal communication), primarily because scientific information on the nutrient dynamics of ADM is currently scarce.

In order to apply manure or other organic amendment to fulfill crop nutrient requirements without increasing the risk of environmental pollution, knowledge of the amendment’s nutrient release characteristics following application is important (Eghball, 2002; Zvomuya et al., 2006). Manure and other organic amendments are a valuable nutrient source when applied to soil at rates consistent with good agronomic practices (Duffera et al., 1999a). Due to the biochemical and physical changes induced by the process of anaerobic digestion, ADM may have different nutrient release patterns relative to raw manure. Results from a recent incubation study (Chiyoka et al., 2011) indicate that ADM-amended soils emit lower N_2O than manure-amended soils, suggesting that changes occurring during digestion can alter microbial processes which control denitrification. It is therefore conceivable that the same digestion-induced changes could alter nutrient mineralization, which is also a microbially-driven process (Eghball et al., 2002).

Past research on nutrient release and availability following organic amendment application has mostly focused on plant available forms (i.e., NH_4^+ and NO_3^-) (Flowers and Arnold, 1983; Bernal and Kirchmann, 1992; Calderon et al., 2005). However, in general, a larger proportion of N (about 50-75% of total N) is in organic form (Havlin et al., 2005), which is only slowly mineralized to available forms. Mineralization of organic N forms is therefore a prerequisite for plant uptake (Chadwick et al., 2000) and the amount of N mineralized from organic sources during the growing season can provide a significant proportion of plant N needs (Eghball, 2000). To utilize ADM efficiently as a nutrient source for crop production, nutrient release following ADM application must therefore be characterized to optimize agronomic benefits and avoid soil N accumulation, which may result in environmental contamination through nitrate leaching and denitrification (Chadwick et al., 2000).

Several incubation (Enwezor, 1976; Whalen et al., 2001; Burger and Venterea, 2008) and growth room (Chadwick et al., 2000; Zvomuya et al., 2006; Helgason et al., 2007) studies have quantified N and P mineralization following amendment application to soils. By eliminating effects of environmental variables, such as temperature and moisture content, which are important factors driving mineralization (Cabrera et al., 2005; Helgason et al., 2007), incubations and growth room bioassays enable the molecular scale evaluation of N mineralization mechanisms (Eghball et al., 2002). Growth room bioassays have the added advantage of approximating plant root competition with microorganisms for nutrient uptake (Helgason et al., 2007). Therefore, a growth room bioassay, with forage barley as the test crop, was used to characterize the nutrient release characteristics of ADM forms vs. Manure. The specific hypotheses were: (i) N and P mineralization in ADM-amended soils differs from that in raw manure-amended soils, and (ii) the quantity of N and P mineralized increases with increase in N application rate.

4.3. Materials and Methods

4.3.1. Organic Amendments

Raw beef cattle feedlot manure (manure), the separated solids fraction of anaerobically digested manure (SS), and pelletized SS (PSS) were collected from a biogas plant near Vegreville in Alberta, Canada. The pellets were cylindrical and approximately 9 mm long with a diameter of 4 mm and a density of 1.2 Mg m^{-3}. Because anaerobic digestion reduces mostly the C concentration of manure, with little if any change in N and P concentrations (Bernal and Kirchmann, 1992; Loria and Sawyer, 2005), pelletization and the concomitant dewatering of SS significantly reduce product volume thereby improving its ease of handling and storage and increasing the economic haulage distance of nutrients to nutrient-deficient cropland (Hammac et al., 2007).

Amendment subsamples were freeze-dried, finely ground (<0.15 mm) and analyzed for total N and C concentration (Table 4.1) by dry combustion (NS-2000 Nitrogen Analyzer, Leco Corporation, St. Joseph, MI). Total P concentration was determined colorimetrically at a wavelength of 880 nm (ascorbic acid method, Murphy and Riley, 1962) by a discrete autoanalyzer (Easy-Chem Pro, Sistea Scientific, LLC, Oak Brook, IL) following acid digestion (Parkinson and Allen, 1975). Inorganic (NO_3-, NO_2- and NH_4-) N concentration was determined on wet amendment samples (0.5 g) (phenate method, Searle, 1984) using a Model AA3 autoanalyzer (Bran+Luebbe, Nordersted, Germany) following extraction with 2 M KCl (30 mL) (Keeney and Nelson, 1982).

4.3.2. Soil Properties

Surface soils (0-15 cm) were collected from two sites in Alberta, Canada: a Dark Brown Chernozem (fine-loamy, mixed, Typic Haploboroll) from a site near Lethbridge (49°42’N; 112°47’W) and a Black Chernozem (silty clay Typic Haplocryoll) from a site near St Albert (53°42’N; 113°47’W). The soils were air-dried, sieved through a 10-mm sieve and stored at room temperature until the start of the experiment. Sub-samples ground to pass through a 2-mm sieve were analyzed for particle size distribution by the pipette method after pre-treatment to remove soluble salts, organic matter, and carbonates (Gee and Bauder, 1986), and for inorganic N content (Table 4.1) as described above for amendments following extraction of soil (5 g) in 2 M KCl (25 mL). Soil inorganic P concentration was determined with an autoanalyzer (as described for total P) after extraction of 1.0 g soil (dry weight) with 20 mL of 0.5 M $NaHCO_3$ (Olsen et al., 1954).

Soil pH and EC were measured in a 1:2 soil: water (mass/volume) suspension using Accumet AB and Accumet AB30 meters, respectively (Fisher Scientific, Hampton, NH). Initial and final (after 5th harvest) soil total N and total P concentrations as well as inorganic N concentrations of soils from 5th harvest were determined as described for the organic amendments. Soil moisture content at field capacity was determined under a suction of 30 kPa using a 500 kPa pressure plate moisture extractor (Model 1600, Soil Moisture Equipment Co., Santa Barbara, CA).

Table 4.1 Selected physical and chemical properties of soils and amendments used in the growth room bioassay.

Property†	Dark Brown Chernozem	Black Chernozem	Raw Manure	SS‡	PSS§
Total N (g kg^{-1})	2.2	7.1	18	15	15
Total C (g kg^{-1})	22	80	355	450	438
C:N ratio	10	11	20	31	29
Total P (g kg^{-1})	0.5	1.1	4.0	3.1	4.3
C:P ratio	42	78	89	145	102
PO_4-P (mg kg^{-1})	38	65	-	-	-
NH_4^+-N (mg kg^{-1})	5	8	3493	2433	217
NO_3^--N¶ (mg kg^{-1})	8	34	<12	<12	<12
pH	6.3	6.3	7.8	8.7	7.6
EC (dS/m)	0.17	0.24	11	5.0	7.0
Moisture content (g kg^{-1})	-	-	628	745	91
Clay (g kg^{-1})	294	451	-	-	-
Silt (g kg^{-1})	271	480	-	-	-
Sand (g kg^{-1})	435	70	-	-	-

† Moisture content, soil pH, and electrical conductivity (EC) on fresh weight basis; total C, total N, NH_4^+-N, NO_3^--N, C:N ratio, total P and C:P ratio on a dry weight basis.
‡ Separated solids from anaerobically digested manure slurry.
§ Pelletized separated solids.
¶ NO_3^--N concentrations in manure, SS and PSS were below detection limit (<0.2 ppm). This threshold corresponds to <12 mg N kg^{-1} after factoring the dilution used for extraction, i.e., 0.5 g soil extracted in 30 mL of KCl.

4.3.3. Experimental Setup

Raw manure, SS and PSS were mixed thoroughly with 1.5 kg of air-dried soil at rates equivalent to 400 and 800 mg N kg^{-1} soil. Assuming 50% availability of total N applied and bulk densities of 1.1 and 1.0 Mg m^{-3} for the Dark Brown and Black soils, respectively, the lower rate is equivalent to 100 kg N ha^{-1} (the standard recommendation for beef cattle feedlot manure application on forage barley in western Canada). A synthetic fertilizer comprising monoammonium phosphate and urea (UMP) and non-amended soil (control) treatments were included for comparison. Monoammonium phosphate was added to supply 34 mg P kg^{-1} (the standard fertilizer P application rate for forage barley in western Canada) and urea was added to achieve total N rates of 400 and 800 mg N kg^{-1}. Triplicate replication was used resulting in a total of 72 units in a split-split plot treatment layout with soil as main plot, amendment as subplot and rate as sub-subplot).

Because amendments were applied to supply the same amount of total N, amounts of P applied varied with amendment. At the 400 mg N kg^{-1} rate, raw manure, SS, and PSS supplied 78, 80, and 128 mg P kg^{-1} soil, respectively, which were doubled at the higher rate. For the UMP treatment (both N rates), monoammonium phosphate was applied at 34 mg P kg^{-1}.

Amended soils were put into 2.25 L plastic pots and watered with reverse osmosis (R.O.) water to bring the soil moisture to field capacity (FC). The pots were then placed in a controlled-environment growth room. After 3 d, the potted soils were seeded to forage barley (cv. Xena*)* at a rate of 15 seeds pot^{-1}. Seedlings were thinned to five per pot after germination. The controlled environment chamber was maintained at a day/night temperature regime of 22/15°C and corresponding photoperiods of 16 h light/8 h dark throughout the study, with humidity maintained at ~60%. Soon after setup, each pot was weighed to provide a baseline weight for moisture replenishment. Throughout the experiment, pots were weighed every other day and watered as needed to bring the soil moisture back to FC.

All pots received initial full-strength nutrient solutions from which N and P were omitted (Zvomuya et al., 2006). The nutrients were added at rates (kg^{-1} soil) of 100 mg K as KCl; 20 mg S as $MgSO_4.7H_2O$; 4 mg of each of Zn as $ZnCl_2$ and Fe as Fe.EDTA; 2 mg Mn as $MnCl_2.4H_2O$; 0.4 mg Mo as $NaMoO_4.2H_2O$, and 1 mg each of B as H_3BO_3 and Cu as $CuSO_4.5H_2O$.

Barley plants were harvested 6 wk after planting by cutting the shoots at the soil surface using a scalpel. Each pot was emptied into an aluminum pan and the roots carefully separated from soil, except for the finer roots that were difficult to separate. The shoots and roots were dried at 60°C for 48 h. Soil from each pot was thoroughly mixed, and a 20-g (dry weight) subsample taken and air-dried. The remaining soils were re-potted and stored in the controlled environment chamber for 24 h, after which macro- and micronutrients were applied at half the rate used during the first planting (Zvomuya et al., 2006). The soils were then re-seeded to forage barley as described previously. This cycle was repeated four times, resulting in 5 crop cycles over a 210 d. From the third crop cycle onwards, ten pre-germinated seedlings were transplanted to each pot and thinned to five seedlings after 3 to 4 d. Pre-germination was implemented to avoid the non-uniform germination observed during the first two crop cycles.

4.3.4. Plant Analysis

Plant shoot and root samples were weighed after oven drying at 60°C for 48 h. The samples were ground to pass through a 0.15 mm sieve using a household grinder for determination of total N and P as described above.

4.3.5. Biomass Yield and Nitrogen and Phosphorus Uptake and Mineralization

Biomass yields from each pot were summed over the 5 growth cycles. Total N and P uptake values were determined for each treatment as illustrated for N:

$$NU = (DW_s \times NU_s) + (DW_r \times NU_r) \qquad [1]$$

where NU and DW are N uptake (mg N kg^{-1} soil) and dry matter yields (g), respectively, and subscripts s and r denote shoot and root material. For each treatment, total N uptake values from the 5 harvests were summed to give the cumulative nitrogen uptake (CNU) (mg kg^{-1} soil) over the 210-d period.

Amendment derived N and P were calculated (using N as an example) as:

$$AmNU = (NU_a - NU_c)/ M_s \qquad [2]$$

where AmNU is amendment-derived N uptake (mg N kg^{-1}), NU_a and NU_c are total plant N uptake (mg N) in amended and non-amended soils, respectively, and M_s is the mass of soil (kg).

The amount of N mineralized from the organic amendments (manure, SS, and PSS) ($Org.N_{min}$, mg N kg^{-1}) was calculated as:

$$Org.N_{min} = (AmNU - N_c)/ M_s \qquad [3]$$

where N_C is the amendment inorganic N that was applied per pot and the subscripts are as defined above.

Apparent N recovery (ANR, %) was calculated as:

$$ANR = (AmNU/TN_{amend}) \times 100 \qquad [4]$$

where TN_{amend} is the total amendment N applied. Apparent P efficiencies were estimated using Eq. 4 and replacing N with P.

4.3.6. Statistical Analysis

Biomass yield, CNU, Cumulative P uptake (CPU), AmNU, amendment-derived P uptake (AmPU), $Org.N_{min}$, ANR, apparent P recovery (APR), residual soil total N, inorganic N and total P concentration data were analyzed using PROC MIXED in SAS (SAS Institute Inc., 2008). Soil, amendment and rate were fixed effects while replicate and combinations of replicate with any of soil, amendment and rate were random effects. For biomass and residual soil N and P data, statistical analysis was performed for amended treatments only (controls excluded) to allow separation of rate effects, i.e., 0 (control) from 400 and 800 mg N kg^{-1} on yields and residual nutrient concentrations, respectively. Treatment differences were considered significant if $P < 0.05$ using the Tukey-Kramer method.

4.4. Results

4.4.1. Cumulative Biomass Yield

Total biomass yield from the five harvests varied with amendment type ($P < 0.0001$); however, amendment effects varied with soil and rate, as indicated by the significant three-way interaction (Table 4.2). In the Dark Brown Chernozem and averaged over the two amendment rates, manure and UMP resulted in similar biomass yields (mean = 29 g N kg^{-1} soil), which were significantly greater than those from SS (26 g N kg^{-1} soil) and PSS (11 g N kg^{-1} soil) applications (Fig. 4.1). By comparison, biomass yields in the Black Chernozem were similar for manure, SS, and UMP (mean = 36 g N kg^{-1} soil), which out-yielded PSS (24 g N kg^{-1} soil) (Fig. 4.1). For all amendments, biomass yields were significantly higher in the Black Chernozem than in the Dark Brown Chernozem. Similarly for non-amended soils, the biomass yield was higher in the Black Chernozem (25 g kg^{-1} soil) than the Dark Brown Chernozem (16 g N kg^{-1} soil).

Biomass yield, averaged over the two soils, increased with increasing rate for all amendments except PSS (Fig. 4.2). At both amendment rates, biomass yields were similar for manure, SS, and PSS and significantly lower for PSS. Control treatments also produced greater biomass yields than PSS-amended soils (Fig. 4.2).

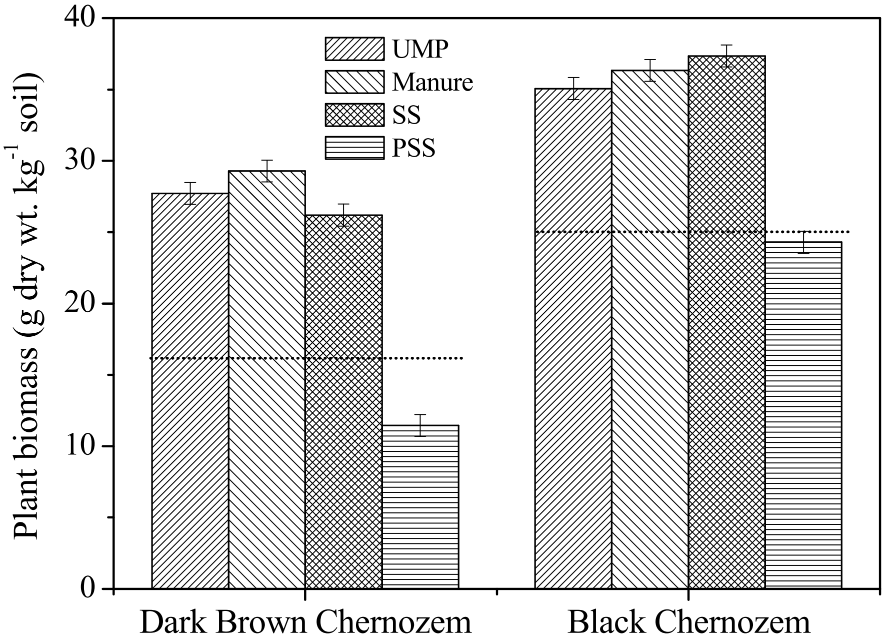

Figure 4.1 Cumulative biomass from five cycles of forage barley grown in Dark Brown and Black Chernozemic soils; amended with raw feedlot cattle manure, separated solid fraction of anaerobically digested feedlot manure (SS), pelletized SS (PSS), and monoammonium phosphate plus urea (UMP). Biomass produced in the control treatments was not included in the statistical analysis and is shown as dotted lines for comparison.

Table 4.2 Total cumulative biomass yield; N, and P uptake; amendment-derived N and P uptake, mineralized organic N, apparent N and P recovery, and residual soil N and P. †

Effect	Cumu. Biomass	CNU	AmNU	Org. N_{min}	Org. N_{min}	CPU	AmPU	ANR	APR	Res. soil N	Res. soil P
	$g\ kg^{-1}$	mg N kg^{-1}			%	mg P kg^{-1}		%		g kg^{-1}	
Soil											
Dark Brown	24	285	147	31	10	50	22	25	32	2.5	0.6
Black	33	541	153	44	12	74	21	25	27	7.4	1.2
Amendment											
Manure	33	460	232	130	31	65	26	38	22	4.9	0.9
SS	32	413	140	55	12	67	27	23	22	5.1	0.9
PSS	18	274	-61	-73	-11	52	10	-10	5	5.0	1.0
UMP	31	504	289	-	-	64	23	49	70	4.7	0.8
Rate (mg N kg^{-1})											
0	-	314	-	-	-	48	-	-	-	-	-
400	26	414	100	21	10	62	15	25	26	4.8	0.85
800	31	510	200	54	12	76	28	25	34	5.0	0.94
P-value											
Soil (S)	<0.0001	<0.0001	0.66	0.39	0.46	0.0002	0.77	0.97	0.003	<.0001	<.0001
Amendment (A)	<0.0001	<0.0001	<0.0001	0.001	0.001	<.0001	0.02	<0.0001	<.0001	0.0003	0.02
S × A	0.0003	0.0004	0.006	0.01	0.19	<.0001	<.0001	0.01	0.001	0.56	0.60
Rate (R)	<0.0001	<0.0001	0.003	0.06	0.54	<.0001	0.01	0.99	0.005	0.002	0.003
S × R	0.02	0.36	0.23	0.34	0.51	0.07	0.003	0.24	0.0004	0.04	0.23
A × R	<0.0001	<0.0001	0.001	0.01	0.95	<.0001	0.0004	0.46	0.002	0.34	0.12
S × A × R	0.89	0.04	0.61	0.34	0.98	0.0001	0.06	0.73	0.003	0.17	0.66

† Cumu. Biomass, cumulative biomass yield; CNU, cumulative N uptake; AmNU, amendment-derived N uptake; Org. N_{min}, mineralized organic N; % Org. N_{min}, percentage of total organic N mineralized; CPU, cumulative P uptake; AmPU, amendment-derived P uptake; Res. Soil N and P, residual total N and P in amended soils after 5 harvests.

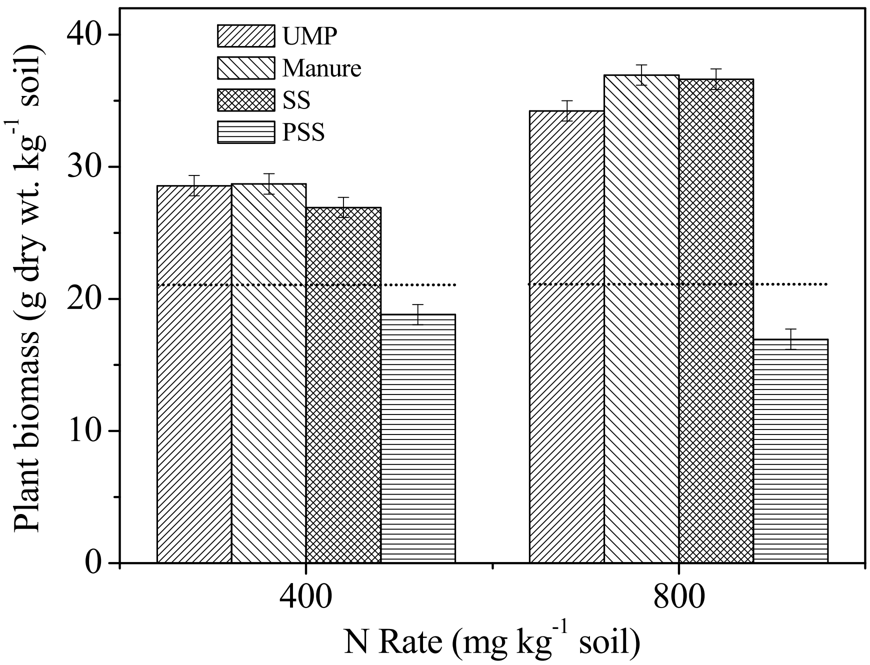

Figure 4.2 Cumulative biomass from five cycles of forage barley grown in amended Dark Brown and Black Chernozems. Amendment treatments are manure: raw feedlot cattle manure; SS: separated solid fraction of anaerobically digested feedlot manure; PSS: pelletized SS; and UMP: monoammonium phosphate plus urea (UMP). Biomass produced in non-amended soils was not included in the statistical analysis and is shown as dotted lines for comparison.

4.4.2. Plant Nitrogen Uptake

4.4.2.1. Cumulative N uptake. All amendments except PSS caused an increase in CNU relative to non-amended soils (Fig 4.3); however, this effect varied with soil type and rate as indicated by the significant amendment x soil x rate interaction (Table 4.2). In the Dark Brown soil, CNU decreased in the order: UMP (405 mg N kg^{-1}) > manure (330 mg N kg^{-1}) ≈ SS (272 mg N kg^{-1}) > PSS (166 mg N kg^{-1}) (Fig. 4.3a). At the lower rate, CNU was similar for manure and SS, but was higher in manure- than SS-amended soils at the higher rate. In the Black Chernozem and at both rates, manure and UMP had similar N uptake values, which were higher than those for SS and PSS. However, by comparison, N uptake was similar for manure and SS (301 mg N kg^{-1}) at the lower rate. Similar to the Dark Brown Chernozem, N uptake in the SS-treated Black Chernozem was greater than that for PSS. Nitrogen uptake in non-amended soils was higher in the Black (441 mg N kg^{-1}) than the Dark Brown (186 mg N kg^{-1}) Chernozem.

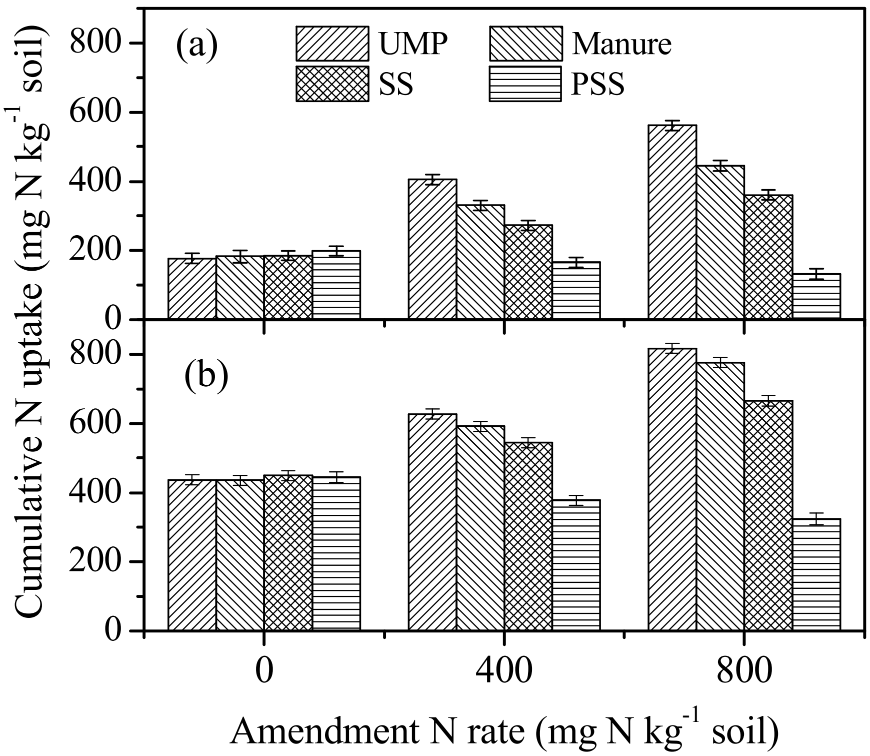

Figure 4.3 Cumulative N uptake by 5 cycles of forage barley grown in (a) a Dark Brown Chernozem and (b) a Black Chernozem. Amendment treatments were: non-amended (control), raw beef cattle feedlot manure (manure), the separated solid fraction of anaerobically digested beef cattle feedlot manure (SS), pelletized SS (PSS), and monoammonium phosphate plus urea (UMP).

4.4.2.2. Amendment-derived nitrogen. Amendment-derived N uptake in both soils decreased in the order UMP > manure > SS > PSS, but the difference between UMP and manure was not significant in the Black Chernozem (Fig. 4.4). All amendments resulted in positive AmNU values except PSS, which depressed plant N uptake relative to both non-amended soils. Amendment-derived N uptake values from manure and SS application were significantly higher in the Black Chernozem than in the Dark Brown Chernozem. Conversely, plants grown in the Dark Brown Chernozem took up more UMP-derived N than those grown in the Black Chernozem. Net N immobilization from PSS application was greater in the Black Chernozem than in the Dark Brown Chernozem.

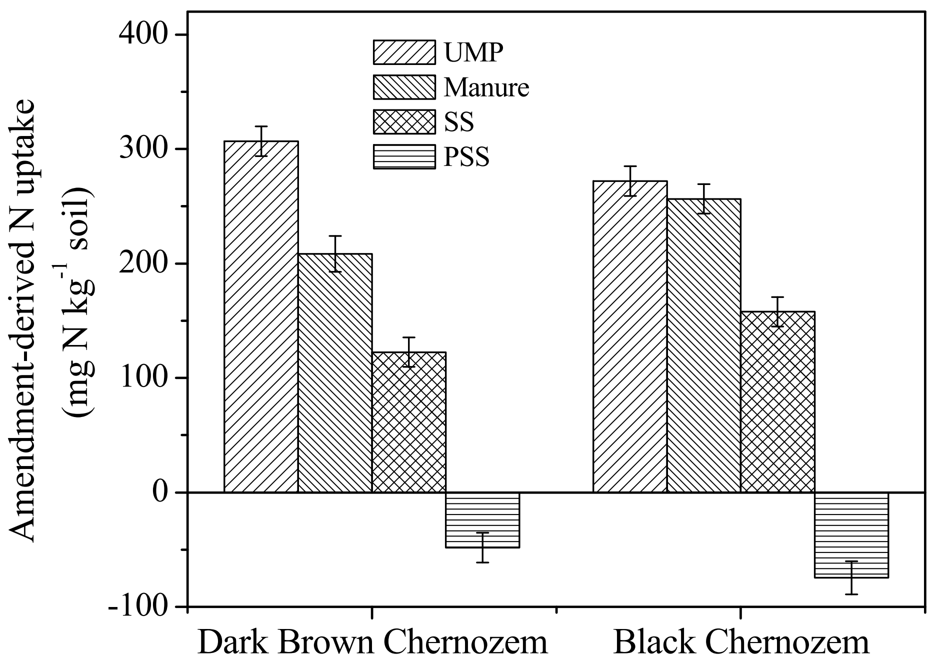

Figure 4.4 Amendment-derived N uptake of forage barley grown in Chernozemic soils amended with raw beef cattle feedlot manure, separated solid fraction of anaerobically digested beef cattle feedlot manure (SS), pelletized SS (PSS), and a synthetic fertilizer comprising monoammonium phosphate and urea (UMP).

For all amendments but PSS, AmNU (mean of the two soils) was higher at the 800 mg N kg^{-1} rate than at the lower amendment rate (Fig. 4.5). In soils amended with PSS, there was net N immobilization, which was higher at the higher application rate. At both rates, N uptake from amendment application decreased in the order: UMP > manure > SS > PSS.

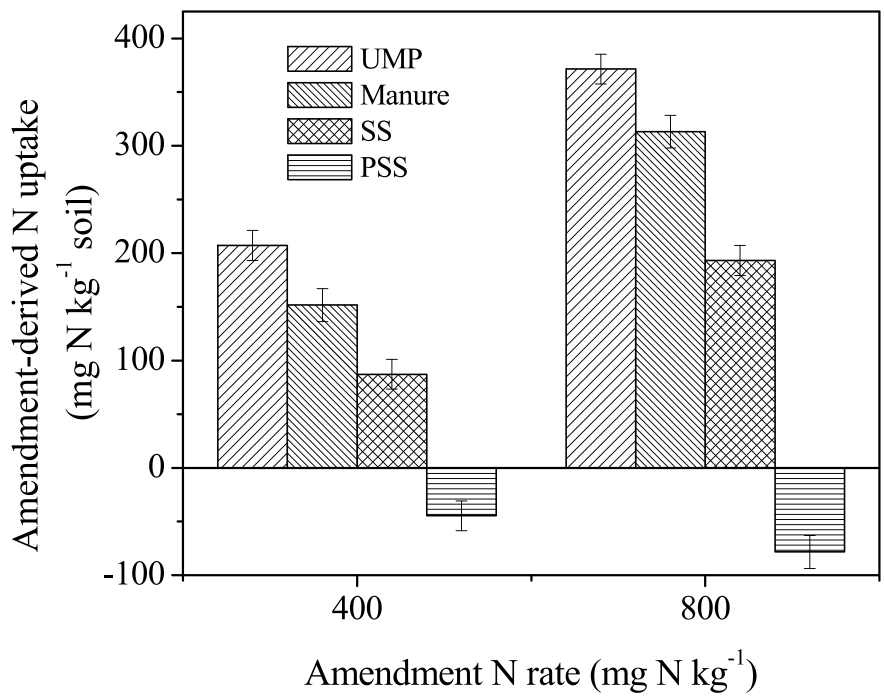

Figure 4.5 Amendment-derived N uptake of forage barley grown in Chernozemic soils amended with raw feedlot cattle manure, separated solid fraction of anaerobically digested feedlot manure (SS), pelletized SS (PSS), and monoammonium phosphate plus urea (UMP), at 400 and 800 mg N kg^{-1} soil rates.

4.4.2.3. Mineralized organic N

Similarly to net N uptake, organic N mineralization in both soils (Fig. 4.6) and at both amendment rates (Fig. 4.7) decreased in the order: manure > SS > PSS, with PSS again resulting in the net immobilization of soil inorganic N. Organic N mineralization from SS application was similar for the two soils whereas manure application resulted in greater mineralization in the Black Chernozem than the Dark Brown Chernozem (Fig. 4.6). Organic N mineralization increased with increasing manure and SS rates but decreased with increasing PSS rate (Fig. 4.7). Net inorganic N immobilization (i.e., negative mineralization) from PSS application was higher in the Black Chernozem than the Dark Brown Chernozem (Fig. 4.6) and at the 800 mg N kg^{-1} rate than at the lower rate (Fig. 4.7).

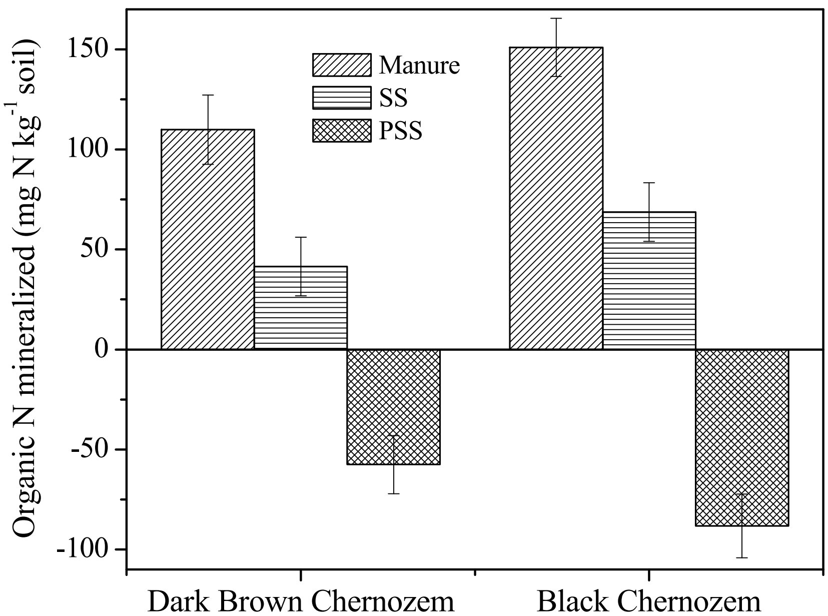

Figure 4.6 Organic N mineralized during a 210-d forage barley bioassay (5 harvests) in Dark Brown and Black Chernozems amended with raw feedlot cattle manure, the separated solid fraction of anaerobically digested feedlot manure (SS), and pelletized SS (PSS).

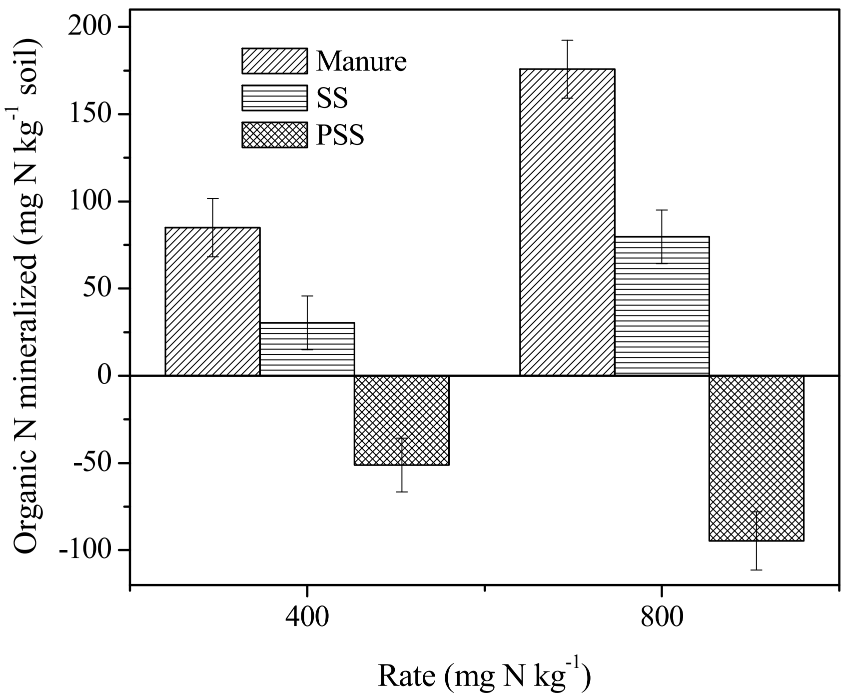

Figure 4.7 Organic N mineralized in Chernozemic soils amended with feedlot cattle manure, the separated solid fraction of anaerobically digested feedlot manure (SS), and pelletized SS, at 400 and 800 mg N kg^{-1} rates in a 210-d growth room bioassay of forage barley.

4.4.2.4. Apparent nitrogen recovery

Overall, plants grown in UMP-amended soils took up the highest fraction of applied N (mean = 49%) while none of the PSS-added N seemed to have been taken up by plants receiving this amendment (-10%) (Table 4.2; Fig. 4.8). In the Black Chernozem, ANR was similar for manure and UMP applications while UMP resulted in a higher ANR than manure in the Dark Brown Chernozem. In both soils, apparent N recovery (mean of the two rates) was greater in manure- (38%) than SS-amended (23%) soils.

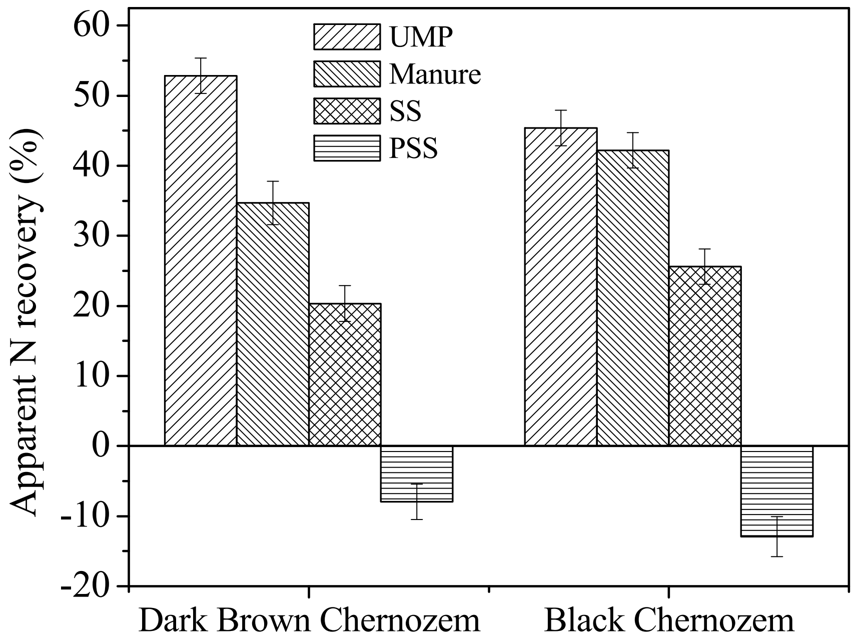

Figure 4.8 Apparent N recovery by five cycles of forage barley grown in Chernozemic soils amended with raw cattle feedlot manure, separated solid fraction of anaerobically digested feedlot manure (SS), pelletized SS (PSS), and monoammonium phosphate plus urea (UMP).

4.4.3. Plant Phosphorus Uptake

4.4.3.1. Cumulative P uptake

Cumulative P uptake differed among amendments ($P < 0.0001$) (Table 4.2) to extents that depended on both soil type and rate ($P = 0.0001$ for the soil × amendment × rate interaction). Plants grown in non-amended soils extracted 35 and 60 mg P kg^{-1} from the Dark Brown and Black Chernozems, respectively (Fig. 4.9). Cumulative P uptake was higher, relative to non-amended soils, for all amendments except PSS, which resulted in P uptake values similar to the non-amended soil in the Dark Brown soil (Fig. 4.9). Doubling the amendment rate from 400 mg N kg^{-1} caused significant increases in P uptake only in the manure and SS treatments in the Dark Brown Chernozem (Fig. 4.9a) and in all treatments in the Black Chernozem (Fig. 4.9b). Manure and SS had similar P uptake values across soils and rates. The SS and manure treatments had higher cumulative N uptake than PSS in both soils and at both rates except the 400 mg N kg^{-1} rate in the Black Chernozem where cumulative P uptake was similar for the three amendments.

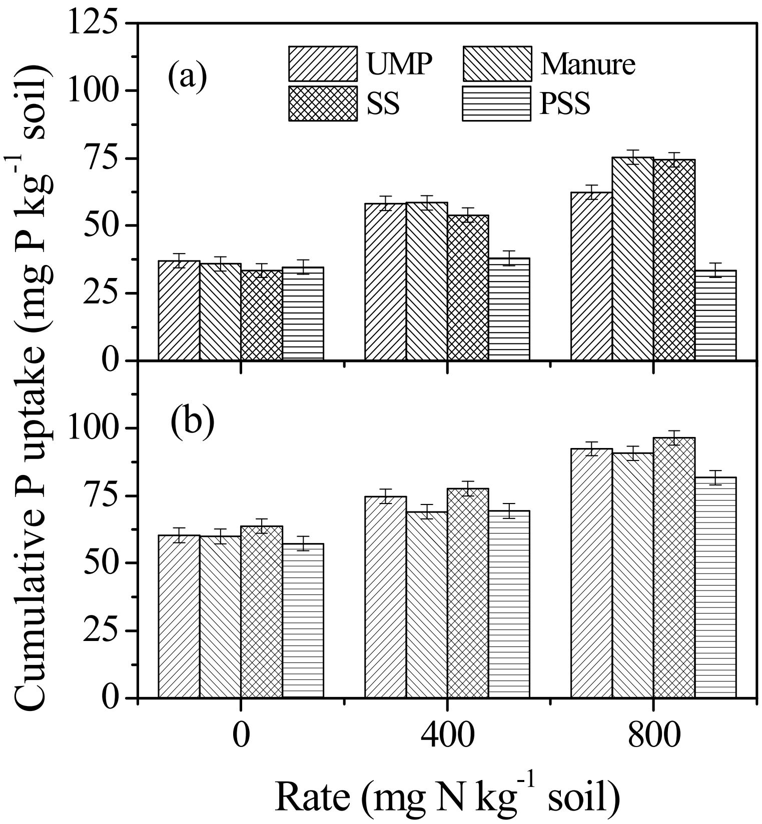

Figure 4.9 Cumulative phosphorus (P) uptake by 5 cycles of forage barley grown in (a) a Dark Brown Chernozem and (b) a Black Chernozem. Amendments were: non-amended (control), raw feedlot cattle manure, separated solid fraction of anaerobically digested feedlot manure (SS), pelletized SS (PSS), and monoammonium phosphate plus urea (UMP), at 400 and 800 mg N kg^{-1} soil rates.

4.4.3.2. Amendment-derived P uptake

Amendment-derived P uptake in the Dark Brown Chernozem was similar for manure and SS and greater for these two amendments than UMP and PSS (Fig. 4.10). By comparison, AmPU uptake was similar for all amendments in the Black Chernozem. Manure and SS applications resulted in greater AmPU values in the Dark Brown than Black Chernozem.

Amendment-derived P uptake, averaged over the two soils, was greater for manure, SS, and UMP than PSS at the 400 mg N kg^{-1} rate, (Fig. 4.11). At the higher rate, AmPU was higher in soils amended with SS and manure than those amended with UMP and PSS. In both soils, AmPU uptake was significantly higher at the higher amendment rate (Table 4.2). At the 400 mg N kg^{-1} rate, APR was higher in the Dark Brown Chernozem than the Black Chernozem for all amendments except PSS. At the higher rate, the Dark Brown Chernozem had lower UMP- and PSS-derived APR but similar manure- and SS-derived APR relative to the Black Chernozem.

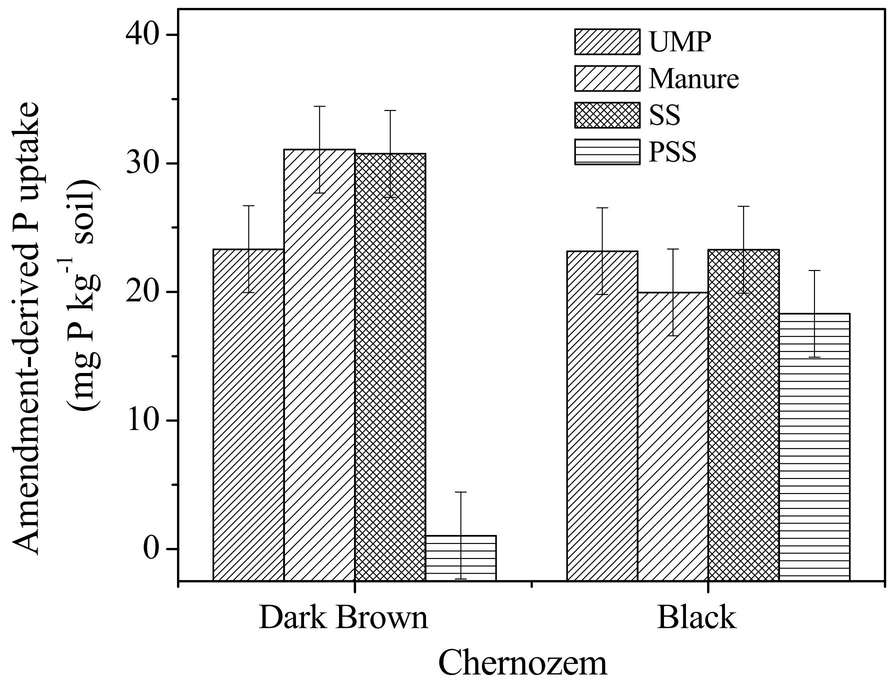

Figure 4.10 Amendment-derived phosphorus uptake by 5 cycles of forage barley grown in Chernozemic soils amended with raw feedlot cattle manure, separated solid fraction of anaerobically digested feedlot manure (SS), pelletized SS (PSS), and monoammonium phosphate plus urea (UMP).

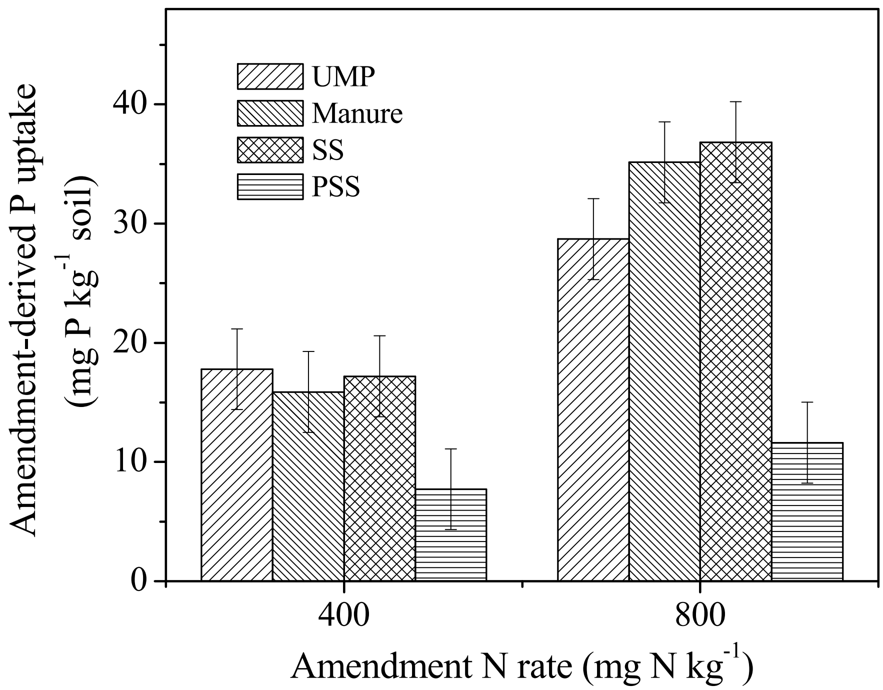

Figure 4.11 Amendment-derived P uptake by forage barley grown in Chernozemic soils amended with raw feedlot cattle manure, separated solid fraction of anaerobically digested feedlot manure (SS), pelletized SS (PSS), and monoammonium phosphate plus urea (UMP), at 400 and 800 mg N kg^{-1} soil rates.

4.5. Apparent P recovery

There was a significant soil × amendment × rate interaction ($P = 0.003$) for APR (Table 4.2). Soils amended with UMP had the highest APR, which increased with increasing rate in both soils, but to a greater extent in the Black Chernozem (Fig 4.12). Manure and SS application resulted in higher APR (mean APR = 28%) than PSS (1%) at both rates in the Dark Brown Chernozem (Fig. 4.12a). In the Black Chernozem, the three organic amendments had similar APR at the lower rate while PSS had a significantly lower APR at the 800 mg N kg^{-1} rate (Fig. 4.12b).

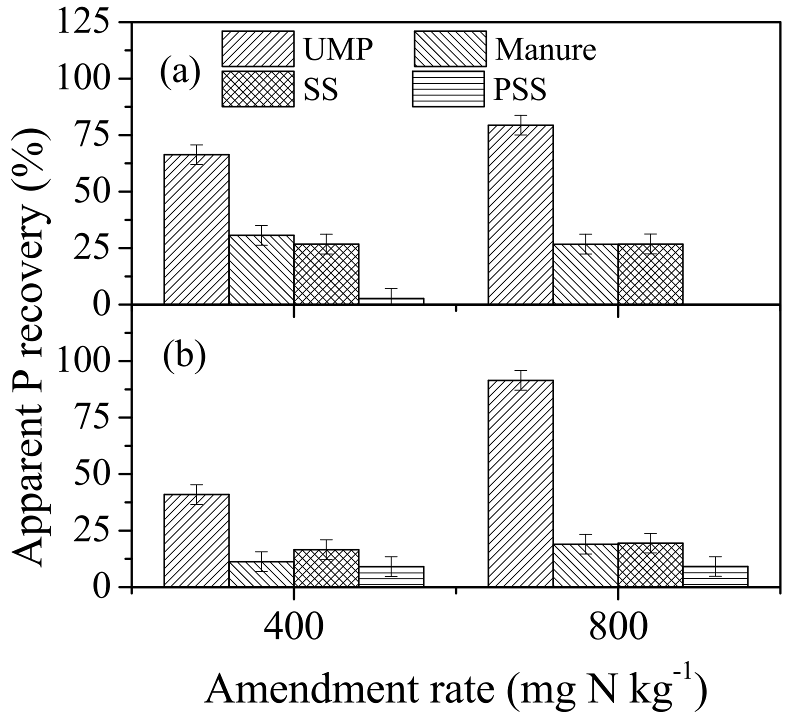

Figure 4.12 Apparent recovery of total applied phosphorus by forage barley grown in (a) a Dark Brown Chernozem and (b) a Black Chernozem amended with raw beef cattle feedlot manure, separated solid fraction of anaerobically digested beef cattle feedlot manure (SS), pelletized SS (PSS), and monoammonium phosphate plus urea (UMP) at 400 and 800 mg N kg^{-1} rates.

4.5.1. Residual Soil Nitrogen

Total N remaining in the amended soils after five harvests (Table 4.2) was influenced by soil ($P<0.0001$), amendment ($P = 0.003$), and rate ($P = 0.0019$). The interaction between soil and rate effects was significant ($P = 0.0443$). Residual N was higher for SS- and PSS-amended soils (mean = 5.1 g N kg^{-1}) than manure- (4.9 g kg^{-1}) and UMP-amended soils (mean = 4.7 g N kg^{-1}). Across amendments, total N increased with increasing rate in both soils. Averaged across amendments and rates, residual soil N was higher in the Black (7.4 g kg^{-1}) than the Dark Brown Chernozem (2.5 g kg^{-1}). Mean residual total N concentrations for non-amended Dark Brown and Black Chernozems were 2.2 and 7.1 g kg^{-1}, respectively.

Residual inorganic (NO_3^- + NH_4^+) N concentrations in amended soils after five harvests differed with amendment type ($P = 0.002$) (Appendix IV). There was a significant soil × amendment interaction effect on residual inorganic N concentration (Fig. 4.13). In the Dark Brown Chernozem, UMP and SS had similar and higher soil inorganic N than PSS, while manure had inorganic N which was similar to SS but lower than UMP. However, in the Black Chernozem, manure had the highest mineral N concentration, which was similar to that in SS-treated soil. Mineral N was similar for UMP and SS treatments while lowest in PSS-amended soil. Mean inorganic N concentrations for control treatments were 67 mg N kg^{-1} for the Dark Brown Chernozem and 62 mg N kg^{-1} for the Black Chernozem.

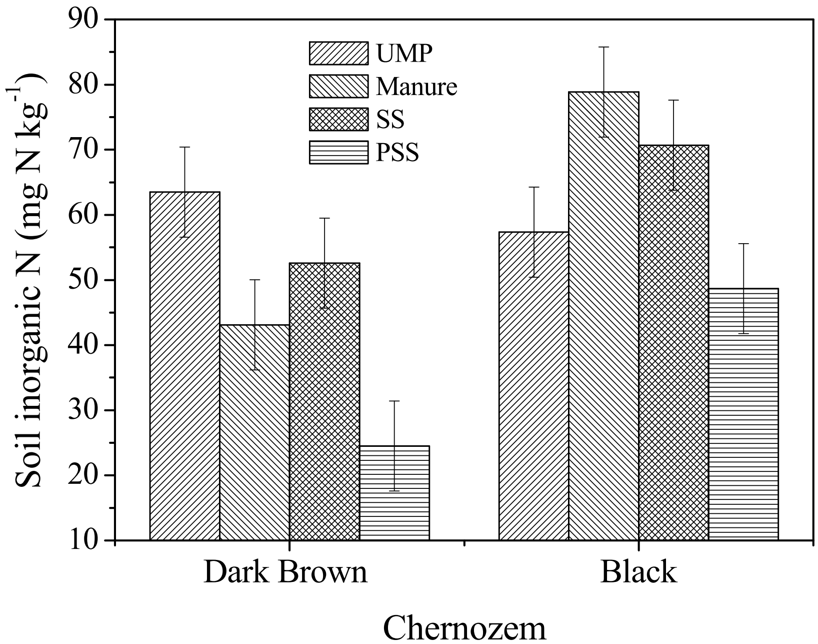

Figure 4.13 Residual soil inorganic N concentration after five harvests of barley in Dark Brown and Black Chernozemic soils amended with raw beef cattle feedlot manure, separated solid fraction of anaerobically digested beef cattle feedlot manure (SS), pelletized SS (PSS), and monoammonium phosphate plus urea (UMP).

The Black Chernozem had higher total P concentration (1.2 g P kg^{-1}) than the Dark Brown Chernozem (0.6 g kg^{-1}). As with residual total N, soil P concentration increased with increasing amendment rate (Table 4.2).

Although residual soil total C varied with amendment ($P = 0.0006$), amendment effects depended on soil type and rate, as indicated by the significant three-way interaction (Appendix). Overall, soil total C was higher with organic amendment (manure, SS, and PSS; mean = 55 g C kg^{-1}) than UMP (51 g C kg^{-1}) application, and increased with increasing amendment rate (Appendix).

4.6. Discussion

4.6.1. Biomass Yield

The lower cumulative biomass in PSS- than UMP-, manure-, and SS-amended soils suggests that nutrient availability from the pellets was a limiting factor for optimal growth. Biomass yield increased with increasing UMP, manure, and SS rates likely due to the higher available nutrient contents at the higher rate. The yield, however, decreased with increasing PSS rate.

4.6.2. Plant Nitrogen Uptake

Mineralization or immobilization of organic N in soil is a function of the decomposability of C compounds in the amendment (Bernal and Kirchmann, 1992; Hammac et al., 2007). Results from the present study show that although SS adequately supplied N to plants, manure- and UMP-derived N was more readily available, allowing greater N uptake from these amendments. Lower N mineralization (AmNU and ANR) in SS and PSS relative to manure can be attributed to the loss of readily available N during processing (Eghball, 2000), i.e., solid-liquid separation and, in the case of PSS, pelletization. Like composting, solid-liquid separation of ADM produces a solid fraction (SS) that is more chemically stable than manure because most of the easily mineralizable N is converted to inorganic forms during anaerobic digestion (Kirchmann, 1991; Bernal and Kirchmann, 1992; Loria and Sawyer, 2005) and partitioned to the liquid fraction during separation.

Differences among manure, SS, and UMP were greater for cumulative N uptake than biomass yield likely due to luxury consumption of N in the manure and UMP treatments (Eghball and Power, 1999). Biomass and N uptake increased with increasing manure, SS, and UMP rates, suggesting greater N availability with higher N applications as observed in other studies (Duffera et al., 1999b). Higher N uptake in fertilizer- vs. manure-amended soils (as observed for the Dark Brown soil), has been reported in other studies (Hammac et al., 2007; Helgason et al., 2007). However, other studies (Hamilton and Sims, 1995; Duffera et al., 1999b) have reported similar or even greater N uptake from organic amendments compared with fertilizer, as observed for the Black Chernozem in the present study, indicating the influence of both soil and amendment characteristics on N availability.

The depression in biomass yield and N uptake observed in PSS-amended soils relative to the control suggests that microbial N immobilization out-competed plant N uptake in these treatments. The loss of labile and readily available N and C compounds during pelletization of SS probably left recalcitrant constituents that were not easily degradable by soil microbes. A similar depressive effect of pelletization on yields and N uptake has been reported for broiler litter (Hammac et al., 2007).

Nitrogen immobilization following application of organic amendments has been widely reported. For example, beef and dairy manure-amended soils showed immobilization during the early stages of a 199-d bioassay in which N uptake by ryegrass was used to estimate mineralization (Chadwick et al., 2000). Beauchamp (1986) reported net N immobilization followed by net mineralization in soils amended with solid beef cattle manure in both field and greenhouse studies where three manures were compared with urea as sources of N for corn (*Zea mays* L.). Ndayegamiye and Isfan (1991) also reported a persistent immobilization effect with composts obtained after 0 and 12 mo of composting, which is consistent with the recommendation to apply additional N fertilizer to hasten decomposition when such composts are applied to sustain plant growth during the year of application (Mamo et al., 1999; Helgason et al., 2007).

The percentage of organic N mineralized increased with increasing initial NH_4-N concentration of the amendment. This is in agreement with Chadwick et al. (2000) who reported a positive relationship between the initial inorganic N concentration of manure and the percentage of organic N mineralized following application of manure to soil in a growth chamber experiment. These results suggest that mineral N rather than organic N is more important for synchronous N availability and plant uptake. The low percentages of total organic N mineralized from SS (12%) and PSS (-11%) relative to manure (31%) indicate that organic N release from amendments with initially low inorganic N concentrations is slow. In fact, none of the organic N from PSS had mineralized after 5 growth cycles. These results are comparable with mineralized organic N values reported by Eghball (2000) for feedlot manure (21%) and composted feedlot manure (11%) applied to corn.

Since the organic N pool of amendments represents a large fraction of total N (80-99% in the present study), it is important to provide producers with reliable recommendations on the N supply from the organic amendments. Current recommendations in western Canada are based on 25-30% of the organic N in livestock manure being available in the first year application (The Prairie Provinces' Committee on Livestock Development and Manure Management, 2011). This guideline works well for raw manure but not for SS and PSS which, in the present study, had less than 25% of organic N mineralized during the bioassay, suggesting that guidelines for raw manure application may not be suitable for its anaerobic digestion derivatives.

Apparent N recovery was greater for manure than SS and PSS, indicating that N release and availability decreased with processing and increasing C:N ratio. Previous studies indicate that net mineralization following manure application can be expected for amendments with a C:N ratio ranging from 16 to 20 (Beauchamp and Paul, 1989; Calderon et al., 2005; Enwezor, 1976; Havlin et al., 2005). Results from the present study are in partial accord with this threshold. Manure with a C:N ratio of 20 had positive AmNU (net mineralization) relative to the control. Despite having a C:N ratio > 20, SS also had net N mineralization, a finding that supports other studies which report a C:N ratio of 25-30 as the threshold for net N mineralization following application of organic amendments (Fox et al., 1990; Trinsoutrot et al., 2000). These contradictory findings regarding the threshold for net N mineralization indicate that the C:N ratio on its own cannot accurately predict N mineralization patterns as reported by Kumar and Goh (2003) and Jensen et al. (2005).

4.6.3. Plant Phosphorus Uptake

Total CPU and AmPU were similar for UMP, manure, and SS, suggesting that manure is as good a P source as synthetic fertilizer and that anaerobic digestion does not alter P mineralization. These results are consistent with Zvomuya et al. (2006) who reported similar CPU by canola (*Brassica napus*) grown in a Dark Brown Chernozemic clay loam soil amended with composted and non-composted cattle feedlot manure, and Eghball and Power (1999) who found similar P uptake by corn for fertilizer, manure, and compost treatments in a field study. Total P uptake and biomass yield were also similar for fescue (*Festuca arundinacea* Schreb) grown in soils amended with poultry litter compost and triple superphosphate in a 103-d growth room study (Sikora and Enkiri, 2005). Plants grown in non-amended soils extracted 35 and 60 mg P kg^{-1} from the Dark Brown and Black Chernozems, respectively suggesting that there was no net mineralization of P from the soils during the bioassay since CNU values were similar to initial available P contents of the two soils (Table 4.1).

Although Hammac et al. (2007) reported lower cumulative P uptake by Italian ryegrass (*Lolium multiflorum* Lam.) and sorghum-sudangrass (*Sorghum bicolour L.* Moench) grown in soil amended with pelletized broiler litter vs. $CaHPO_4$, biomass yield was similar for these amendments, indicating that pelletized broiler litter can serve as a viable P source for plants. However, in the present study, pelletization of SS depressed P mineralization and uptake by plants (APR = 9%), which, along with depressed yields relative to the control, suggests that the P in PSS is not readily available for plant uptake.

Plant recovery of P from raw manure and SS (mean = 22%) was lower than that from UMP (70%), indicating that raw manure and SS have lower P supply capacity than synthetic fertilizer, a finding in accord with Motavalli et al. (1989) but contradictory to Eghball et al. (2005). The P added with manure and SS (118 mg P kg^{-1}) and PSS (192 mg P kg^{-1}) was much higher than that added with UMP (34 mg P kg^{-1}), indicating that P release from organic amendments is much slower than that in synthetic P fertilizer (Zvomuya et al., 2006).

Increasing manure and SS rates to 800 mg N kg^{-1} resulted in greater P uptake, affirming similar findings from other studies (Duffera et al., 1999b; Sikora and Enkiri, 2005). Interestingly, in the UMP-amended Black Chernozem, increasing N but not P rate resulted in a significant increase in P uptake, suggesting greater P availability in the soil likely due to faster organic matter decomposition at higher fertilizer N rate.

Nitrogen-based organic amendment application may result in significant build-up of P in the soil (Eghball and Power, 1999) because the N:P ratios of most manures are usually smaller than the N:P needs of most crops (Gilbertson et al., 1979). Although the N:P ratios of amendments used in the present study (4.4, 4.9, and 3.6 for manure, SS, and PSS, respectively) were higher than the 2.6 and 1.9 values for beef cattle feedlot manure and composted manure, respectively, reported by Eghball et al. (1997), P build-up could occur, especially with application of PSS which had the highest P content but lowest APR coupled with N immobilization. Application of high PSS rates to supply sufficient inorganic N and minimize N immobilization may cause greater P accumulation in soil, increasing the risk of environmental P pollution.

4.6.4. Soil Residual Nitrogen

The higher residual total N concentration in SS- and PSS- than manure-amended soils is likely the result of lower N mineralization coupled with higher organic N concentrations in the SS- and PSS- than manure-amended soils. The readily-available N in UMP treated soils allowed for greater plant uptake resulting in lower residual total N concentration compared with the organic amendments which had most of their N in the organic form. Lower residual inorganic N in PSS than other organic amendments is likely due to the recalcitrant nature of organic N in the PSS relative to manure and SS which had higher organic N mineralization. Similar to initial soil conditions, the Black Chernozem had higher residual mineral N, total N and total C than the Dark Brown Chernozem, reflecting the differences in the N and C economies of these soils (Table 4.1).

4.7. Conclusions

Although raw manure and SS had similar biomass yields, plant net N uptake was higher in manure than SS treatments, likely due to slower organic N mineralization and lower initial NH_4-N concentration in SS compared to raw manure. Despite the benefits of pelletization, application of PSS caused net N immobilization and depressed yields relative to non-amended soils. While N mineralization (AmNU and ANR) increased with increasing manure and SS rates, N immobilization increased with increasing PSS rate. Although manure, SS, and UMP had similar AmPU, which increased with increasing amendment rate, P was more readily available in UMP than in the organic amendments. Application of SS and PSS, at higher rates than manure or additional N fertilizer with SS and PSS is recommended to expedite organic N release and ensure adequate available N for plant uptake, especially early in the growing season.

4.8. References

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5. GENERAL SYNTHESIS

The importance of knowledge regarding nutrient mineralization following application of organic amendments, for both agronomic and environmental reasons is indisputable (Eghball et al., 2002; Cabrera et al., 2005). Anaerobically digested beef cattle feedlot manure is a nutrient-rich substrate cogenerated during biogas production from anaerobic digestion of beef cattle feedlot manure. The ADM is applied at rates used for raw beef cattle feedlot manure despite changes occurring during anaerobic digestion, which could conceivably alter nutrient release dynamics and N_2O emission when the ADM is applied to soil. Two laboratory incubations and a growth room bioassay of forage barley (*Hordeum vulgare* L.) were conducted to test this hypothesis using two common soils in western Canada (a Dark Brown Chernozem and a Black Chernozem); four amendments [raw beef cattle feedlot manure (manure), separated solids fraction of ADM (SS), pelletized SS (PSS), and a synthetic fertilizer comprising urea and monoammonium phosphate (UMP)], and two rates (400 and 800 mg N kg^{-1}).

Application of SS resulted in lower N_2O emissions compared to raw manure. This was likely due to lower NH_4-N concentration and slower organic N mineralization in SS- than manure-amended soil, as shown by the N mineralization patterns (Chapters 3 and 4). Compared to manure, SS thus supplied lower inorganic N for nitrification and denitrification, the two main N_2O-producing processes in soil (Havlin et al., 2005). Surprisingly, cumulative N_2O emission values for PSS- and manure-amended soils were not significantly different. The high N_2O emissions from PSS-amended soil can be explained by the high concentration of N in the pellets which probably created hot spots for nitrification and denitrification. By comparison, SS was homogenously mixed with soil and did not have high N concentration spots. Increasing manure, SS, and PSS rates resulted in higher N_2O emissions but this increase was only significant for manure treated soils.

Anaerobically digested manure and raw manure had different nutrient release patterns. At the end of the 70-d incubation, soils amended with SS had lower available N than soils amended with raw manure, and PSS-amended soils had lower available N than SS treatments. Although anaerobic digestion of livestock manure has been reported to increase inorganic N concentration (Bernal and Kirchmann, 1992; Kirchmann, 1991; Loria and Sawyer, 2005), solid/liquid separation of the digestate partitions most of the available N into the liquid fraction, leaving the SS with lower available N concentrations than the raw manure. Also, ADM is characterized by the presence of energy-rich and easily decomposable C, which is favourable for N assimilation by microorganisms (Bernal and Kirchmann, 1992) hence the greater extent of N immobilization in SS- than manure-amended soils. Pelletization further lowers dissolved N concentration in SS so that application of PSS to soils caused greater N immobilization than SS. These findings are in accord with the lower apparent crop N recoveries of SS and PSS compared with manure in the bioassay study.

The lower biomass yield from PSS compared to SS and manure indicates that slow nutrient release from PSS was a limitation for optimal plant growth in this treatment. While biomass yield increased with increasing manure and SS rates likely due to greater nutrient availability at higher than lower rates, it decreased with increasing PSS rate. Higher PSS amounts probably caused greater N immobilization, diminishing the plant-available N pool. Although SS supplied enough N to sustain plant growth, manure- and

UMP-derived N was more readily available as shown by the higher plant N uptake in the latter treatments. Consequently, N use efficiency was higher in manure (38%) than SS (23%) and PSS (-10%).

All amendments resulted in net P mineralization throughout the incubation period without any P immobilization (i.e., greater inorganic P concentration in amended than control soils). At the end of the 70-d incubation, manure and SS had similar available P. These results are consistent with the similar net P mineralization values from the bioassay for manure and SS [cumulative P uptake (CPU) and amendment-derived P uptake (AmPU)], and indicate that anaerobic digestion does not alter P availability for plant uptake. Therefore, if applied at equivalent total N rates, raw manure and SS will supply similar plant-available P, a situation that increases the potential for soil P accumulation and risk of P loss to surface and groundwater (Eghball, 2002) should SS be applied at higher rates than raw manure to improve N supply capacity of SS. In both the incubation and bioassay studies, PSS application resulted in lower P mineralization compared to manure and SS suggesting that PSS-P is not readily available for plant uptake.

Effect of higher amendment rate on mineralized N depended on soil and amendment type. Higher manure application resulted in higher N mineralization only in the Black Chernozem while greater N immobilization at the higher rate was observed for manure-, SS-, PSS- treated Dark Brown Chernozem and for SS-and PSS-amended Black Chernozem. Similarly, changes in CPU with increasing PSS rate also depended on soil type; increasing at higher rate in the Black Chernozem but decreasing at higher rate in the Dark Brown Chernozem. These results reiterate the importance of both soil and amendment properties on N and P mineralization-immobilization-turnover and hence net mineralization.

5.1 Conclusions and Recommendations

Overall, the incubation- and bioassay studies showed that SS and PSS have different N and P mineralization and N_2O emission characteristics compared to raw manure following application to agricultural soils. Effects of soil type and amendment rate on N and P mineralization and N_2O emission were evident.

Given the lower N mineralization potential of organic N in SS and PSS than manure, it may be prudent to supplement these amendments with mineral N fertilizer to improve N mineralization rate hence optimizing crop yields. Application of SS and PSS at higher N-based rates, though an alternative option to address this limitation of low N release from ADM forms, may not be favourable because of the increased potential for P accumulation and risk of loss to the environment since these amendments, particularly SS, had mineralized P and plant P uptake that were similar to those of raw manure.

Although pelletization is beneficial for nutrient alleviation in concentrated livestock production areas, application of PSS depressed biomass yields and N mineralization relative to non-amended treatments, and significantly increased N_2O emissions relative to SS. Fortifying the PSS-pellets with mineral N may improve nutrient mineralization. However, this could also further increase N_2O emission when the N-fortified pellets are applied to soil. Further research on the effects of fortifying pellets with mineral N is therefore needed.

The use of controlled-environment experiments eliminated effects of variable factors such as soil moisture and temperature, and hence provided an insight into N and P mineralization mechanisms following application of SS and PSS vs. raw manure. However, extrapolation of results from these studies to field conditions should be done with caution because of the use of an artificial environment as well as soil disturbance.

5.2 References

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6. APPENDICES

Appendix I: Pictures of Organic Amendments and Barley from the Second Crop Cycle

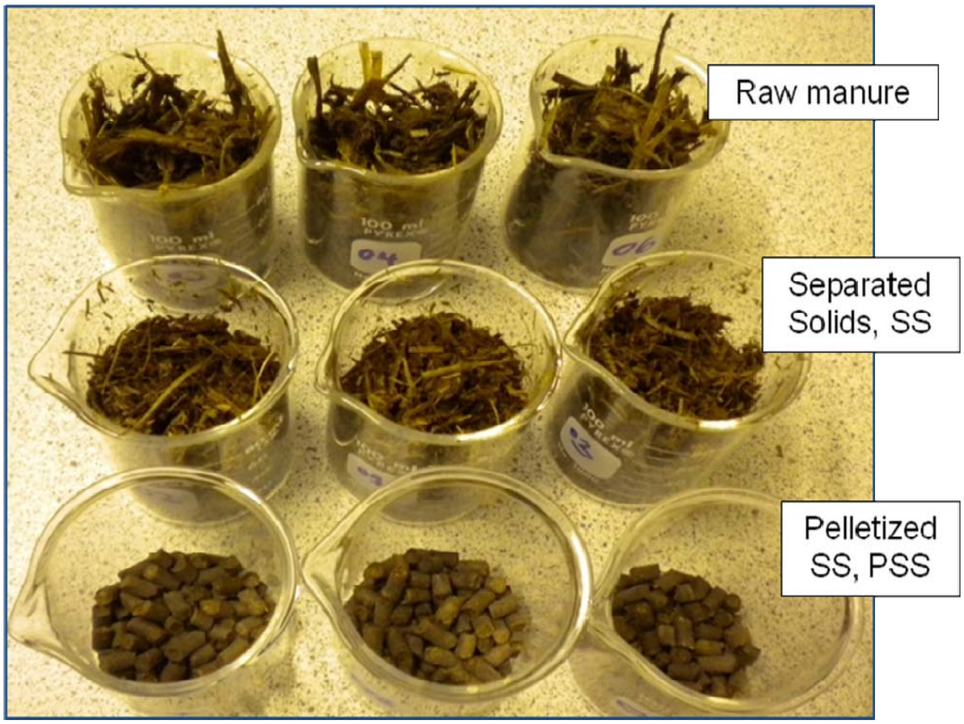
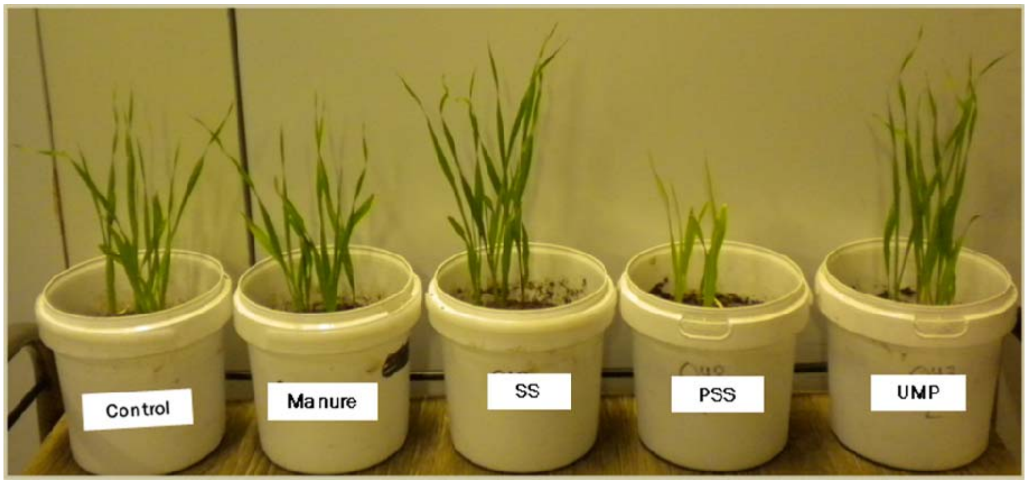


Plate 1: Raw beef cattle-feedlot manure, separated solids of anaerobically digested beef cattle feedlot manure (SS), and pelletized SS (PSS).



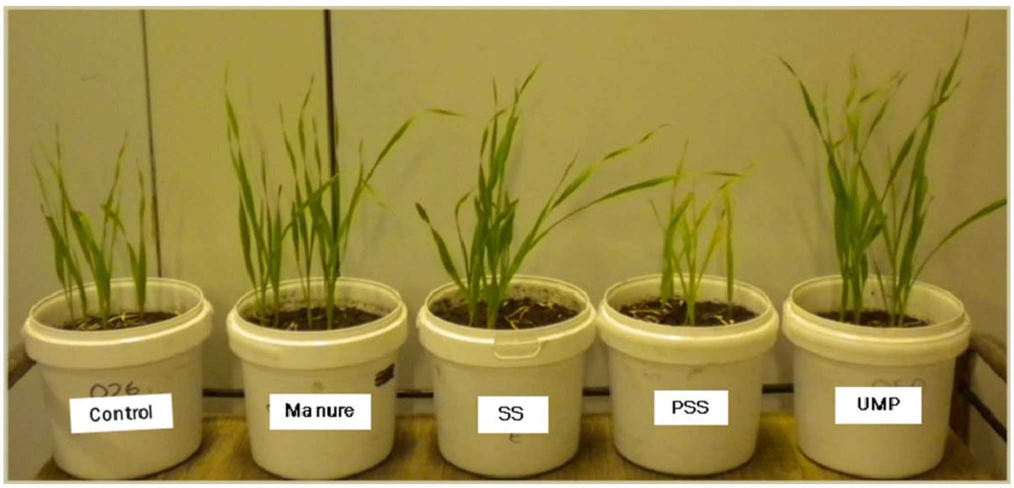
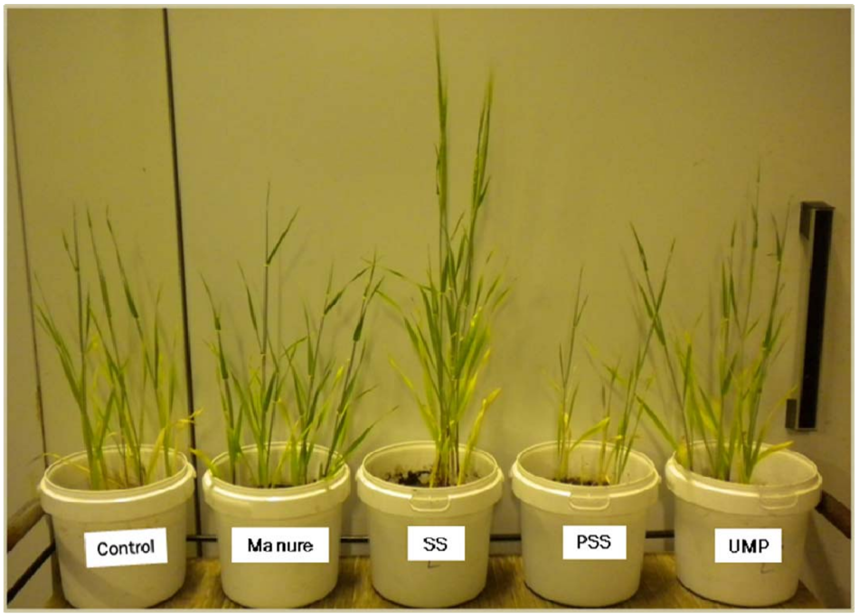


Plate 2: Crop cycle 2 barley plants 16 d after seeds were sown. Top picture shows plants grown in the Dark Brown Chernozem and the bottom picture shows plants grown in the Black Chernozem.



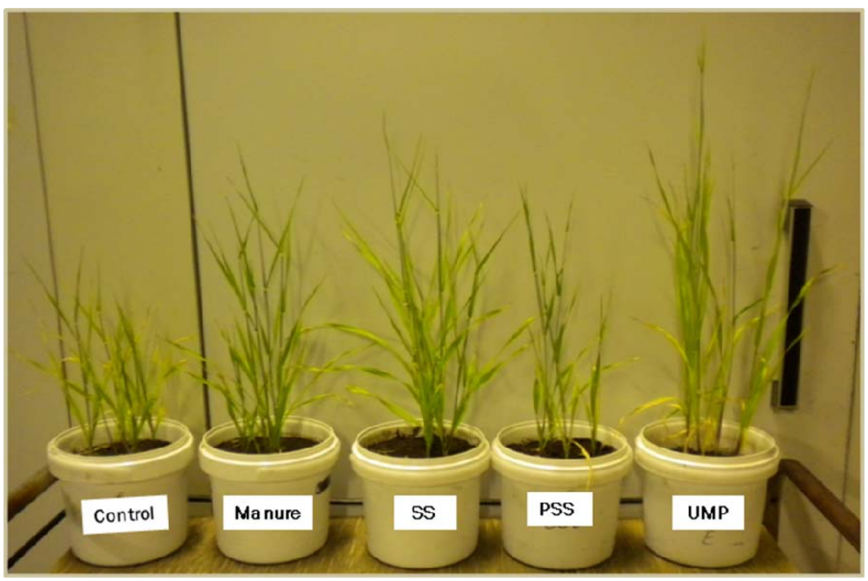


Plate 3*:* Crop cycle 2 barley plants 31 d after seeds were sown. Top picture shows plants grown in the Dark Brown Chernozem and the bottom picture shows plants grown in the Black Chernozem.

Appendix II: Supplementary Analysis of Variance (ANOVA) Tables

Table A1. Analysis of variance for concentrations of residual inorganic nitrogen (N) and total carbon in amended soils

Effect	**Residual soil inorganic N**†	**Residual soil total C**
	mg N kg^{-1}	g C kg^{-1}
Soil		
Dark Brown	46	26
Black	64	83
Amendment		
Manure	61	53
SS	62	56
PSS	37	57
UMP	60	51
Rate (mg N kg^{-1})		
400	56	53
800	54	56
P-value		
Soil (S)	0.10	<.0001
Amendment (A)	0.002	0.0006
S × A	0.002	0.30
Rate (R)	0.67	<.0001
S × R	0.52	0.10
A × R	0.27	0.08
S × A × R	0.31	0.04

† Inorganic N is the sum of NO_3^- + NH_4^+.

Table A2. Analysis of variance for concentrations of nitrogen and phosphorus in barley shoots and roots

Effect	Nitrogen concentration in shoots	Nitrogen concentration in roots	Phosphorus concentration in roots	Phosphorus concentration in roots
	—— g N kg^{-1} plant ——		—— g P kg^{-1} plant ——	
Soil				
Dark Brown	13	17	2.85	1.75
Black	18	19	2.86	1.74
Amendment				
Manure	16	19	2.48	1.71
SS	15	19	2.62	1.81
PSS	15	16	3.68	1.77
UMP	17	19	2.65	1.69
Rate (mg N kg^{-1})				
400	16	19	2.81	1.75
800	15	18	2.91	1.74
P-value				
Soil (S)	<.0001	<.0001	0.97	0.74
Amendment (A)	0.001	0.002	<.0001	0.26
S × A	<.0001	0.12	0.41	0.15
Rate (R)	0.17	0.03	0.21	0.84
S × R	0.00	0.04	0.04	0.85
A × R	0.17	0.22	<.0001	0.01
S × A × R	0.48	0.82	0.15	0.03

† Least squares means generated from analyzing concentrations of N and P in shoots and roots (mean concentrations for the 5 crop cycles of barley).