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Measuring the Influence of Bedding on Soil Nitrogen Losses and Corn Crop Nitrogen Characteristics for Fall Applied Solid Beef Cattle Manure in Eastern South Dakota

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This thesis is approved as a creditable and independent investigation by a candidate for the Master of Science in Agricultural and Biosystems Engineering degree and is acceptable for meeting the thesis requirements for the degree. Acceptance of this thesis does not imply that the conclusion reached by the candidate are necessarily the conclusion of the major department.

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ABBREVIATIONS

AIC	Akaike's Information Criteria
cm	Centimeter
d	day
g	gram
µg	micro-gram
kg	kilogram
in.	inch
ft.	feet
h	hours
ha	Hectares
in.	inch
Inc.	Incorporated
KCL	Potassium Chloride
m	Meters
mL	milliliter
max	Maximum
min	Minimum
Mg	Mega gram

Mm	millimeter
N	Nitrogen
NH_3	Ammonia
NH_4	Ammonium
NO_3	Nitrate
N_2O	Nitrous Oxide
Oct.	October
SE	Standard Error
SAS	Statistical Analysis Software
SD	South Dakota
SDSU	South Dakota State University
TAN	Total Ammoniacal Nitrogen
USA	United States of America
USEPA	United States Environmental Protection Agency
CO	Colorado

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ABSTRACT

MEASURING THE INFLUENCE OF BEDDING ON SOIL NITROGEN LOSSES AND CORN CROP NITROGEN CHARACTERISTICS FOR FALL APPLIED SOLID BEEF CATTLE MANURE IN EASTERN SOUTH DAKOTA

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Nitrogen (N) is a major component of chlorophyll which plays a key role in the photosynthesis process in crops. The N is one of the highest demanded nutrients by all plants for their growth and reproduction. Manure or inorganic fertilizer is often applied to fulfill the crops' N demand. However, the applied N sources have the potential of N losses in different forms from the soil volume in many ways such as ammonia (NH_3) volatilization, aerial nitrous oxide (N_2O) loss, nitrate (NO_3^--N) leaching, and runoff and/or erosion. Soil fertility, crop yield, water quality, and air quality can be reduced by excessive N losses from the soil volume. The goal of this study was to understand the effect of fall-applied solid beef manure with bedding on nitrogen movement and transformations during corn production. The objectives of the research were to measure the N losses (NH_3, N_2O, and soil water NO_3^--N concentration) from the soil for fall-applied N and corn production, then compare the impact of applied N form (solid beef cattle manure with bedding (MB), solid beef cattle manure only (MO), urea only (UO) and no-fertilizer (NF)), in Brookings County, SD. The methods for collecting samples for soil N losses were semi-static open chambers for NH_3 flux, static chambers for N_2O flux, and suction lysimeters for soil water. The applied N were 130 and 184 kg ha^{-1} in Year 1 and Year 2, respectively. The studied showed the average (±SE) soil NO_3^--N for UO (105 ± 9 kg ha^{-1}) was significantly higher than the remaining treatments; soil NO_3^--N was 72 and 65 kg ha^{-1} for manure treatments MB and MO, respectively. The average (±SE) total soil NO_3^--N for Year 1 (83 ± 6 kg ha^{-1}) was significantly higher than Year 2 (67 ± 5 kg ha^{-1}). However, the average total soil NO_3^--N at Pre-plant stage was significantly higher than V6 and Postharvest stages in both years. The study results did not show any significant difference in total soil NO_3^--N due to interaction of Treatment and Growth Stage. Furthermore, the average NH_3 flux, and N_2O flux were significantly affected by treatments. The highest N_2O flux was produced by the UO (79.0 ± 24.9 μg m^{-2} h^{-1}) plots, whereas the flux released from MB was 49.0 ± 15.1 μg m^{-2} h^{-1} and for MO it was 33.3 ± 10.3 μg m^{-2} h^{-1}. The N_2O flux obtained from UO was significantly higher than NF, while MB and MO-produced N_2O fluxes were not significantly different than neither UO nor NF. The highest NH_3 flux occurred from the MB treatment, which was 3.4 ± 0.9 g ha^{-1} h^{-1}, however this flux was only significantly different than NF. The NH_3 fluxes from UO and MO were not significantly different than MB and NF. The average (±SE) N_2O and NH_3 fluxes for control (NF) were 25 (±8) μg m^{-2} h^{-1} and 1.4 (±0.4) g ha^{-1} h^{-1}, respectively. The average soil water NO_3^--N concentration was not significantly different among the treatments ($P < 0.05$). The average soil water NO_3^--N concentration was significantly greater in Year 1 (12.5 ± 2.0 mg L^{-1}) compared to Year 2 (6.5 ± 2.0 mg L^{-1}). Crop N characteristics such as leaf-N and grain-N tended to be different ($P < 0.1$) among treatments, with a higher N concentration in UO-treated plots. The corn yield was not significantly affected by treatment in Year 1 (the only year measured). The study aids the understanding of soil N losses via various paths and the effect of fall-applied solid manure with or without bedding on soil N losses and N transformations. Overall, the data obtained from our study will be used in model application purposes, which will help to

further understand the factors and processes affecting nutrient transformations and losses during corn production with beef cattle manure.

CHAPTER 1 INTRODUCTION

1.1 Background

Nitrogen (N) is a critical nutrient in the respiration, metabolism, growth, and reproduction systems for plants (Dinnes et al., 2002; Follett and Hatfield, 2001; Ohyama, 2010). Also, N is a major component of chlorophyll, which helps to convert light energy into chemical energy in the photosynthesis process (Havlin et al., 2005). About 78% (by volume) of the atmosphere is N, but it is in inert gas form and not directly available for the plants (Follett and Hatfield, 2001; Havlin et al., 2005; Ohyama, 2010). Nitrogen is found in various forms in soil, such as organic matter, soil organisms and microorganisms, ammonium-N, nitrite-N, and nitrate-N (Bremner, 1965a; Cameron et al., 2013; Lamb et al., 2014). However, the proportion of N in soil is only about 0.1 to 0.6% in the top 15 cm of soil, depending on the soil type (Bremner, 1965b; Cameron et al., 2013). Hence, additional N input is required to fullfill crop N requirements. Between 1950 and 2000, world grain production increased three times from 631 to 1840 million tons due to a significant contribution of N fertilizer (Mosier and Syers, 2004). As the world population continues to increase, crop production must also increase, but there is limited arable land to fulfill the demand (Mosier and Syers, 2004). Increasing crop production in the limited arable land is only possible if N fertilizers are used efficiently while minimizing negative impacts in the surrounding environment (Cassman et al., 2002; Mosier and Syers, 2004; van Grinsven et al., 2015).

Soil and plants gain N from various sources like biological and atmospheric fixation, direct addition of manure and commercial fertilizers, crop residue, and animal tissues (Hoos et al., 2000; Lamb et al., 2014; Petrovic, 1990; Vitousek et al., 1997). The rate of N uptake depends on crop types, soil properties, and weather factors (Ohyama, 2010; Provin and Hossner, 2001). However, not all the forms of N are usable for plants. Mainly two inorganic forms of N in soil, nitrate-N (NO_3^--N) and ammonium-N (NH_4^+-N), can be used by plants via the roots. Organic matter N may covert to usable forms of N through mineralization (Havlin et al., 2005; Robertson and Groffman, 2007). Soil microorganisms play an important role to break down or transform the organic matter N to NO_3^--N and NH_4^+-N form of N (Hart et al., 1994; Schimel and Bennett, 2004).

Soil N is a very important nutrient for crop production, however, nitrogen can be potentially lost from the soil when manure and N fertilizers sources are over-applied or mismanaged (Dinnes et al., 2002; Hatfield and Cambardella, 2001). Soil N can be lost via various processes such as volatilization, denitrification, leaching, runoff, and erosion (Lamb et al., 2014; Loecke et al., 2004; Miller et al., 2009; Paramasivam et al., 2009). Human activities in the agricultural sector are often responsible for potential soil N losses which may pollute air or water quality (EPA, 2006). Globally, in 2005, about 60% of N_2O emission was from agriculture due to anthropogenic activities (IPCC, 2007). Nitrous oxide loss to the atmosphere contrbutes to global warming as well as depletion of the ozone layer (Bouwman et al., 2001; EPA, 2013). Similarly, 20 to 50% of total agricultural ammonia (NH_3) loss is from land applied organic fertilizers (Sintermann et al., 2012; Sommer and Hutchings, 2001). Ammonia is an important environmental pollutant which has had a wide variety of impacts such as soil acidification, acid rainfall, eutrophication of ecosystem (International Fertilizer Industry Association, 2001). Also, when ammonia is released from the soil surface to atmosphere, it reacts with atmospheric gases such as sulfur dioxide or nitrogen oxides (in the presence of water) to form particulate matter less than 2.5 micrometers that is very harmful for human and animal health, and the environment (Bittman et al., 2014; Hodan and Barnard, 2004). Nitrate-N loss via leaching can vary from 5 to 50% of applied N depending on crop type, soil properties, N rate, and climatic condition (Sainju, 2017). The loss of NO_3^--N from agricultural soil is a major contributor for building a dead zone or hypoxia condition in the Gulf of Mexico (Daigh et al., 2015; Goolsby and Battaglin, 2000; Goolsby et al., 2001). The United States Environmental Protection Agency (US-EPA) has set the maximum level of NO_3^--N concentration to not exceed 10 mg NO_3^--N L^{-1} in drinking water (US-EPA, 2002). Therefore, in agriculture, appropriate management practices are required to improve N use efficiency for crops and reduce N losses to the environment.

Various management practices mitigate soil N losses and increase N use efficiency in the soil (Piccini et al., 2016; Shaviv and Mikkelsen, 1993; Tilman et al., 2002). Soil N losses can be minimized by implementing different strategies such as appropriate N application at appropriate time, reducing tillage, crop rotation, using soil tests and plant monitoring, and improving N application technique (Ahmed et al., 2013; Dinnes et al., 2002; Jokela and Randall, 1989; Mallarino and Wittry, 2010; Smith et al., 2007; van Grinsven et al., 2015). Also, integration of these management strategies and plant breeding with higher N use efficiency may contribute to sustainable agriculture systems which may protect and improve soil, water, and air quality (Baligar et al., 2001). Appropriate rates of feedlot manure with bedding application practice may increase soil organic matter, nutrient contents, and improve soil quality and soil productivity (Amiri and Fallahi, 2009; DeLuca and DeLuca, 1997).

Bedding is generally used on animal farms for animal comfort, to reduce animal injury, and aid in manure handling (Bey et al., 2002; Smith and Hogan, 2006). Manure types, bedding types, and application rate of organic amendments can influence N and phosphorus (P) uptake, and soil physical properties such as water flow and water holding capacity compared to inorganic fertlizer (Airaksinen et al., 2001; Miller et al., 2010; Miller et al., 2014; Miller et al., 2009). Miller et al. (2010) found that soil inorganic N, soil P, and soil mineralizable N were significantly affected by manure, but the effects changed with year or bedding or rate of application or their interactions. However, bedding with rich carbon content manure can immobilize N temporarily in the soil, delaying the release of plant-usable forms of N (Crohn, 2004). Later, when microbes decompose carbon, they utilize carbon to generate energy to grow and reproduce and those microbes help N mineralization in the soil (Crohn, 2004). This research will explore the influence of bedded and bedded solid manure on soil nitrate, N losses, crop characteristics (leaf-N and grain-N), and yield.

1.2 Objectives

The overall goal of this research was to measure the influence of bedding on soil N losses and corn crop N characteristics (leaf-N, grain-N and yield) for fall-applied solid beef cattle manure in Eastern South Dakota. The specific objectives were:

(1) To determine the impact of fall-applied bedded and non-bedded solid beef cattle manure, and urea fertilizer on total soil nitrate-N, soil water nitrate-N concentration, crop N and yield.

(2) To assess the influence of solid beef cattle manure with bedding application on soil fluxes of ammonia (NH_3) and nitrous oxide (N_2O) following manure application and during the growing season.

CHAPTER 2 LITERATURE REVIEW

2.1 The role of manure and urea in the nitrogen cycle

Nitrogen (N) in soil transforms into various forms through biological and physical processes (Bierman and Rosen, 2005; Follett and Hatfield, 2001; Lamb et al., 2014). The soil-N cycle shows how the various forms of N move in or out of the soil system (Figure 2.1; Lamb et al., 2014). Nitrogen compounds can be classified into two groups: reactive N and nonreactive N (Follett, 2008). Both groups exist in nature in equilibrium through the balanced process of the N cycle. Nonreactive N includes inert N_2 gas and organic N, whereas reactive N includes all inorganic N forms (Follett et al., 2010; Galloway et al., 2008). Most N in the soil is tied up in organic matter which resists being consumed by plants. However, soil microorganisms present in the soil break down the organic forms of N into the plant usable forms (nitrate (NO_3^-)) and ammonium (NH_4^+)) (Havlin et al., 2005; Johnson et al., 2005). Plants uptake the available forms of N from soil via their roots system; however, N uptake depends on plant age and type, environment, and other factors (Havlin et al., 2005).

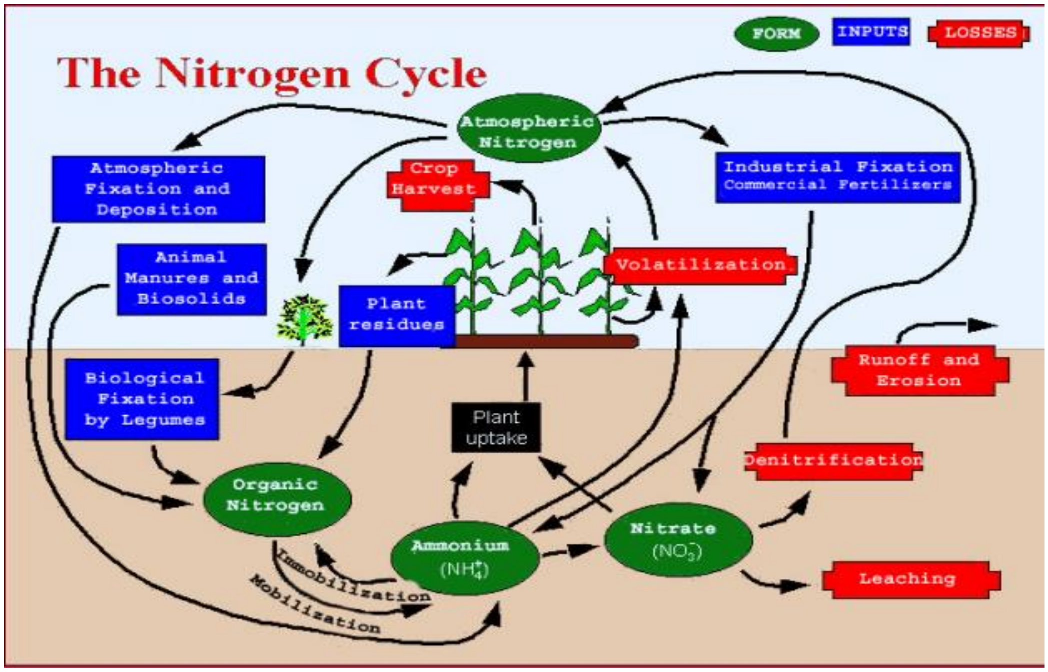

Figure 2.1 The nitrogen cycle (Lamb et al., 2014)

The main N inputs to the soil for plant growth are symbiotic or non-symbiotic fixation, atmospheric fixation, application of animal manure and commercial fertilizer, plant residue, and soil organic matter (Bierman and Rosen, 2005; Follett and Hatfield, 2001; Lamb et al., 2014). Manure applied to the soil contains both reactive N (NH_4^+) and nonreactive N (organic N) (Ketterings et al., 2005). Organic N cannot be used by crops until it converts into inorganic N forms (NH_4^+ or NO_3^-). The soil microbes present in the soil convert organic forms of N into the inorganic N by the mineralization process (Follett, 2008; Havlin et al., 2005; Shober, 2015).

A common form of commercial fertilizer is urea. Urea is hydrolyzed by the enzymatic action of urease, a common enzyme found in soil systems (Mobley and Hausinger, 1989) to ammonia (NH_3), which dissociates in water to exist in equilibrium with NH_4^+. The NH_4^+ is a usable N form for crops. Higher soil pH (>7) favors NH_3, which can be lost to the atmosphere via volatilization (Follett et al., 2010; Follett and Hatfield, 2001; Havlin et al., 2005).

Many human-driven activities such as burning fossil fuels (including burning forests and burning grasslands) and application of N-based fertilizer have a significant impact on the N cycle because these activities can highly increase N in an ecosystem (Bernhard, 2010; Vitousek et al., 1997). Over application or mismanagement of both manure and/or urea in the soil can result in N loss as gaseous or nitrate forms to the environment (Dinnes et al., 2002; Hatfield and Cambardella, 2001). Also, the deposition of reactive N by burning fossil fuels and biomass, application of N, and natural sources of nitrogen oxide (lightening and biogenic soil emissions) may fertilize both terrestrial and marine ecosystems that enhance the carbon storage (carbon sequestration) (Galloway et al., 1994; Maaroufi et al., 2015). The losses of N from the soil volume by volatilization, nitrification or denitrification, leaching, or runoff/erosion may create a problem for the ecosystem. The loss mechanisms are described below (Section 2.1.1 to 2.1.5), and the impacts of these losses are described in Section 2.2.

2.1.1 *Ammonia loss through volatilization*

In the soil solution, total ammoniacal nitrogen (TAN) compounds NH_3 and NH_4^+ exist in equilibrium dependent on pH. The NH_4^+ is stable in solution, but NH_3 ions are subject to loss as a gas to the atmosphere (Equation. 2.1) (Follett, 1995; Follett, 2008).

$$NH_4^+ + OH^- \rightleftharpoons NH_3 + H_2O \qquad (2.1)$$

Soil pH and TAN concentration are important factors which control the magnitude of NH_3 loss to the atmosphere (Follett, 1995). The rate of NH_3 volatilization increases with high soil pH (>7) and high soil temperatures because both factors increase the relative amount of NH_3 concentration in the soil solution (Follett, 1995; Jones et al., 2007; Stevenson and Cole, 1999).

In neutral or acidic soil, NH_3 loss is low for NH_4^+-containing fertilizer (e.g., ammonium nitrate, ammonium sulfate, ammonium phosphate, etc.) compared to NH_4^+ -forming fertilizer (e.g. anhydrous ammonia, aqua ammonia, urea, etc which can form ammonium ion (NH_4^+) after reacting with water) because soil solution pH is not increased while adding NH_4^+ fertilizer (Havlin et al., 2005). However, NH_4^+ -forming fertilizers (urea or urea-containing fertilizer) increase soil solution pH during the hydrolysis reaction (Havlin et al., 2005). The amount of NH_3 loss is also affected by cation excahnage capacity (CEC) (loss is higher in soil of low CEC), soil moisture, soil organic matter, environmental conditions (temperature, wind speed, and precipitation), and management practices (types of N sources, timing and mode of N application, tillage practices) (Al-Kanani and MacKenzie, 1992; Bouwman et al., 1997; Jones et al., 2013; Jones et al., 2007; Ribaudo et al., 2011; Stevenson and Cole, 1999).

2.1.2 *Nitrous oxide loss*

Farmlands are considered a major source of N_2O (Rotz et al., 2012). Nitrification and denitrification are both microbial transformation processes and are responsible for N_2O release from the soil (Andreae and Schimel, 1990; Bremner, 1997; Maag and Vinther, 1996; Rotz et al., 2012). Nitrification is the conversion of ammonium (NH_4^+) into nitrate (NO_3^-) by microbial oxidation and is an aerobic process. Nitric oxide (NO) and N_2O are intermediates in this process (Rotz et al., 2012). Denitrification occurs in soil under anaerobic condition, where the microbial reduction produces NO_2, NO, and

N_2O as intermediates while converting NO_3^- to N_2 (Havlin et al., 2005; Mosier and Klemedtsson, 1994; Rotz et al., 2012). The general reaction of denitrification process (Equation. 2.2) occurring in soil (upward arrows indicate possible N loss as a gaseous form) (Follett, 1995; Havlin et al., 2005) is:

$$NO_3^- \rightarrow NO_2 \text{ (nitrogen oxide)} \rightarrow NO \text{ (nitric oxide)} \rightarrow N_2O \uparrow \rightarrow N_2 \uparrow \quad (2.2)$$

These nitrification and denitrification microbial processes are influenced by oxygen concentration, inorganic N concentration, carbon availability (organic matter), soil properties (soil moisture, soil bulk density, soil pH, soil types, cation exchange capacity), N sources, and climatic factors (air temperature, rainfall, wind, humidity) (Beauchamp, 1997; Dustan, 2002; Havlin et al., 2005; Jarecki et al., 2008; Mathieu et al., 2006; Rotz et al., 2012).

Thapa et al. (2015) observed cumulative N_2O emission and soil inorganic N intensity is linearly correlated. However, Adviento-Borbe et al. (2010) argued that flux N_2O-N should not be interrelated with current N inputs or soil nitrate concentrations, but instead suggested that N_2O variation may be due to long-term effects of animal manure addition and legume rotations on soil structure, labile carbon or microbial communities.

Venterea et al. (2010) conducted research to compare N_2O emission and soil chemical properties for anhydrous ammonia and urea application under corn-corn or corn-soybean rotation in southeastern Minnesota. They found N_2O emission was higher for anhydrous ammonia fertilizer compared to urea in corn-corn and corn-soybean systems compared to urea. Also, they observed that annual N_2O emissions increased while shifting cropping system from corn-soybean to corn-corn. Similarly, Engel et al.

(2010) observed average N_2O loss was 2.0, 2.7, and 5.8 g N kg^{-1} of applied 100 kg N ha^{-1} urea-N through broadcast, band, and nest placements, respectively. Likewise, N application timing affects the N_2O emission from soil. Hao et al. (2001) measured the influence of N fertilizer (ammonium nitrate with 0, 50, 100 or 200 kg N ha^{-1}) application timing and straw/tillage practices on soil N_2O emission under irrigation between 1996 and 1997. They found N_2O emission was higher for fall application of N fertilizer (ammonium nitrate) than spring application. They explained that higher N_2O was produced from fall application fertilizer because before planting, fall-applied N had a long time for denitrification, and freeze-thaw events in the early spring caused greater N_2O fluxes. Also, Hernandez-Ramirez et al. (2009) observed in their study that N_2O emission was strongly influenced by manure application time; but, their result showed N_2O emission in spring injected liquid swine manure was 1.8- and 3.4-fold higher than emissions following fall injection. Lower N_2O fluxes from fall-applied pig slurry manure were associated with cold weather and wet soils (Rochette et al., 2001)

2.1.3 *Nitrate leaching*

Nitrate-N (NO_3^--N) is a major chemical form of N in soil that is taken up by crops (Schuchman, 2010). In soil, negatively charged clay mineral surfaces can repel NO_3^- forms of N because of similar charged ions (Follett, 1995). As a result, NO_3^- ions are highly mobile in soil and tend to leach through the pores of soil particles under the rooting zone when water movement and NO_3^- content are high in soil (Follett, 1995; Grant et al., 2002; Havlin et al., 2005; Provin and Hossner, 2001). The risk of NO_3^--N loss by leaching is a major route in humid climates and under irrigated cropping system (Havlin et al., 2005). However, NO_3^--N leaching below the rooting zone can also occur in semiarid conditions under cultivated systems (Campbell et al., 1984). Several factors may affect the maginitude of NO_3^--N loss via leaching such as: a) rate, time, source, and methods of N fertilization; b) soil profile characteristic which affects percolation; c) amount and time of rainfall and/or irrigation; and d) cropping intensity and crop N uptake (Havlin et al., 2005; Haynes et al., 1986; Janzen et al., 2003; Provin and Hossner, 2001; Schuchman, 2010; Williams et al., 2012).

Nitrate leaching from farmland is a major concern for the environment which is highly influenced by the application of manure and/or fertilizer practice. Basso and Ritchie (2005) conducted 6-years of field study in Michigan to quantify effects on NO_3^--N leaching due to the application of animal manure, compost, and inorganic fertilizer in a maize-alfalfa rotation. They observed a higher amount of NO_3^--N leaching for dairy manure, followed by compost (50% Oak leaves + 50% dairy manure on dry weight), and urea treatment to supply 120 kg of total N ha^{-1} in the fine-loamy soil. Additional studies showed liquid manure application produced higher NO_3^--N leaching than inorganic fertilizer (Ball-Coelho et al., 2004; Elmi et al., 2005).

Bedding in manure also influences NO_3^--N leaching. Land application of a broiler litter (manure and bedding) treatment showed lower NO_3^--N concentration at 1-m depth than commercial fertilizer (ammonium nitrate), but average concentrations for both treatments were less than 10 mg NO_3^--N L^{-1} (Wood et al., 1996). Similarly, Karimi et al. (2017) showed lower NO_3^--N leaching through straw bedded solid pig manure than liquid pig manure, as the straw may immobilize the N in the solid pig manure. Likewise, bedding influences soil properties such as total carbon (C) (Wood et al., 1996), C: N ratio, water-filled pore space, and water-soluble total N, NH_4^+-N, and NO_3^--N (Miller et al., 2014).

Sometimes, the use of one management practice to minimize one form of loss may raise the likelihood of other losses. For example, application of slurry for reducing ammonia loss can increase NO_3^--N leaching in agricultural soil (Powell et al., 2011).

2.1.4 *Surface runoff and soil erosion*

Soil N loss through soil erosion and surface runoff may reduce soil nutrients, impair surface water quality (Lamb et al., 2014), and affect economy and environment (Pimentel et al., 1995; Udawatta et al., 2006). The N loss by soil erosion depends upon the slope of the land, soil texture, amount of soil loss, N content of the soil, conservation practices, and climatic condition. Nitrogen loss by surface runoff varies due to cover crops, source of applied N and timing, rainfall intensity, soil crusting, infiltration capacity of the soil, and soil temperature (Czapar et al., 2008; Knisel, 1980; Pimentel et al., 1995; Ross et al., 2008; Williams et al., 2012). Studies also show that the concentration of NO_3^--N in surface runoff is higher in soil under conventional tillage compare to soil under no-tillage (Follett et al., 2010; McDowell and McGregor, 1984).

Soil erosion by water includes the process of detachment, transport, and deposition of soil particles (Czapar et al., 2008; Foster et al., 1985). During soil erosion, NH_4^+ binds to the surface soil particles and other sediments while NO_3^- is water soluble and thus moves along with water until it reaches surface water bodies.

2.1.5 *Crop nutrient uptake and nutrient removal*

Crop nutrient uptake and nutrient removal are also considered as N loss from the soil volume. Crop nutrient uptake refers to the total amount of nutrients taken by crops throughout the growing season that are contained in different parts of plants like grain, leaves, stalks, and roots, whereas nutrient removal means nutrient removed from the field after harvesting the field crops (Heard and Hay, 2006; Roberts et al., 2015). The amount of nutrient removal from the field crop is less than total nutrient uptake by crops because nutrient contained in the residue (i.e., leaves, stalk, stubble) is returned to the soil (Roberts et al., 2015).

The crop nutrient uptake and nutrient removal rates rely on crop types, crop yield, and soil fertility; however, nutrient uptake by crop varies with soil and climatic conditions (CFI, 2001). For example, low soil moisture, low soil temperature, deposition of excessive lime near the root zone, high soil moisture, nutrient imbalance may limit the crop nutrient uptake (CFI, 2001). Also, removal amount of N with crop yields change as a function of the crop (Robertson, 1997). Binford (2010) found that corn grain yield is the most significant factor in nutrient removal by corn, and the mean N removal concentration was 0.88 kg per hectoliter of grain. Similarly, Canadian Fertilizer Institute (CFI, 2001) presented the average N removed from soil is 175 kg N ha^{-1}, when removing 11.2 tonnes-ha^{-1} corn silage. However, when the yield goal is 0.63 kg m^{-2} (100 bushels/acre), corn grain N uptakes average about 172 kg N ha^{-1} throughout the growing season, and grain harvest from field removes between 98 and 120 kg N ha^{-1} (CFI, 2001).

2.2 Impact of manure (with or without bedding) and inorganic fertilizer

Manure can supply similar plant nutrients as commercial fertilizers while also increasing the organic matter and improving soil quality (Gelderman et al., 2004; Khaleel et al., 1981). Bedding is a complicating factor for determining nutrient availability when it is mixed with manures, because of its absorbency, water holding capacity, nutrient content, and structural integrity properties (Zehnder et al., 2000). These properties of bedding can change the manure properties and soil properties after field application (Miller et al., 2014). In addition, Miller et al. (2017) found bedding significantly affected the soil salinity parameters. Wood-chip bedding can lower soil pH, soluble cations and anions, and electrical conductivity (EC) (Miller et al., 2017). This section describes manure and inorganic fertilizer application effect on N losses and impact of N losses.

2.2.1 *Ammonia volatilization*

Ammonia emission is one of the main causes of low N uptake by crops from animal manure (Paramasivam et al., 2009) or fertilizer applied fields (Bouwman et al., 2002). In many countries, agriculture contributes 20 to 80% of NH_3 emission, wherein livestock manure and N fertilizers are the major contributing sources (Aneja et al., 2008; Jantalia et al., 2012; Misselbrook et al., 2000). Hristov et al. (2011) showed that an average 25 to 50% of N excreted daily from cattle is lost to the atmosphere by volatilization of ammonia (NH_3). Ammonia emission can increase significantly if surface-applied manure or urea is delayed for incorporation into the soil (Ribaudo et al., 2011). It is estimated that immediate incorporation of surface applied solid manure may reduce NH_3 emission by at least 90% (Webb et al., 2010). Paramasivam et al. (2009) conducted a laboratory study to quantify NH_3 emission on the application of different animal manures in fine sand and loamy sand. Their results indicated that solid swine manure produced a greater NH_3 emission compared to poultry manure while applying the same amount of manure (rates for both manure 0, 2.24, 5.6, 11.2, and 22.4 Mg of manure ha^{-1}) and NH_3 loss due to volatilization increased with increasing manure application rate. Cumulative NH_3 volatilization loss over 19 days was 4 to 27% and 14 to 32% of total N for poultry litter manure and swine manure, respectively (Paramasivam et al., 2009). Greater NH_3 loss in swine manure treatment was probably due to the higher total N content in swine manure compared to poultry manure. Jantalia et al. (2012) evaluated NH_3 loss from four urea-based N fertilizers. They observed that following irrigation of 16 to 19 mm of water 1-day after fertilization, the NH_3 loss was between 0.1 and 4.0% of total N from surface-applied N-fertilizers (urea, SuperU, urea-ammonium nitrate, and polymer-coated urea).

Due to human activities in agriculture, NH_3 emission into the atmosphere has been increasing. The U.S. Environmental Protection Agency (EPA) has projected that 50 to 85% of total US human-made ammonia volatilization comes from animal agriculture in the United States (Battye et al., 1994; Gay and Knowlton, 2005). Ammonia loss into the environment has had a wide variety of impacts such as soil acidification, acid rainfall, eutrophication of terrestrial and aquatic ecosystem, and respiratory and cardiovascular problems in humans (Bouwman et al., 2002; Bouwman et al., 1997; Gay and Knowlton, 2005; Krupa, 2003). All these facts indicate that NH_3 loss to the atmosphere produces not only an environment impact but also a risk to human health.

2.2.2 *Nitrous oxide flux*

Nitrous oxide is a potent greenhouse gas occurring in agricultural production which has about 310 times the global warming potential than that of a CO_2 molecule (Dusenbury et al., 2008; EPA, 2013; IPCC, 2014; Rotz et al., 2012) and can destroy stratospheric ozone (Crutzen, 1972, 1974; Schlesinger, 2009). In 2009, U.S. N_2O emissions were 4 million metric tons carbon dioxide equivalent, where agriculture accounted for 73% of total N_2O emission (EIA, 2011). Similarly, the United States EPA Greenhouse Gas Emission Inventory Report in 2016 stated that the 78.9% of total U.S. N_2O emission was contributed by agricultural soil management activities (example: manure and fertilizer application) and cropping practices in 2014. Nitrous oxide emission varied from year to year, and the overall N_2O emissions increased by 5.9 percent between 1990 and 2014 (EPA, 2016). Both reports showed that the agriculture sector is the largest source of N_2O emission in the United States. Globally, about 50% of N_2O flux emits from agricultural soil, caused by human influence mostly due to application of N fertilizer (Grace et al., 2011).

2.2.3 *Nitrate leaching*

Nitrate leaching from agricultural fields impacts crop yield and the environment. Leachate NO_3^--N represents a loss of crop-available nitrogen (Dinnes et al., 2002; Provin and Hossner, 2001). Nitrate-N discharges from the agricultural field can contaminate groundwater and surface waterbodies (lakes and rivers) (Dinnes et al., 2002; Gentry et al., 2007; Randall and Mulla, 2001). The hypoxia condition created in the Gulf of Mexico is an example of excess N discharge into receiving rivers and lakes (Dinnes et al., 2002; Mitsch et al., 2001; Rabalais et al., 2001). Also, an excess of NO_3^--N in the waterbodies

(>10 mg L^{-1}) can significantly affect fresh water and marine animals (Camargo et al., 2005; US-EPA, 2002). Nitrate concentrations in drinking water exceeding 10 mg L^{-1} may pose a risk to pregnant women and human babies (DNR, 2014; US-EPA, 2002).

2.2.4 *Crop nitrogen and yield*

Management practices like manure or fertilizer application timing, rate, types, application methods, and crop rotation can influence the soil available mineral N. The soil available mineral N is directly linked with crop yield. Ultimately, crop yield varies with soil management practices, soil condition, soil N availability, and weather condition. Ahmed et al. (2013) did a field study in central Iowa that determined the effect of liquid swine manure application on soil nutrients, pH, organic matter, and yield. They found the residual soil NO_3^- significantly higher for spring injection of swine manure than fall injection, and corn yield was also significantly higher for spring injection plots. The reduction of yield in fall may be due to excessive leaching of nutrients via soil volume between manure application and corn growing period (Ahmed et al., 2013). Based on short and medium-term aspects, application of commercial inorganic fertilizers is more attractive than manure application due to their convenience, ease of application and handling, and reliable high yield (Hepperly et al., 2009).

2.3 Overview of conservation practices that affect manure and/or urea in the nitrogen cycle

The Midwest is an agriculture dominated landscape, which has a massive impact on environmental quality. Therefore, agricultural producers are often encouraged to adopt conservation practices that help to reduce the impact on the environment caused by agriculture (Prokopy et al., 2014). Conservation practices are implemented to conserve the soil from erosion (wind or water), improve water quality and increase the profits to the producers (Hoag and Osmond., 2012). Currently, several conservation practices are applied by producers in the United States to conserve nutrients in the soil and protect the environment. One of the conservation strategies is application and timing of manure or fertilizer during crop production period because appropriate application of manure and/or fertilizers may improve crop yield and reduce environmental effects. Also, agricultural sustainability will be improved if manure and/or fertilizer are managed carefully. Nutrient management is a major concern while applying manure and/or fertilizer in soil. Therefore, the 4R concept (right source, right rate, right time, and right place) is an approach for nutrient management in the soil that can help to increase nutrient use efficiency, enhance the agricultural productivity, profitability, sustainability, and protect the environment (Bruulsema et al., 2009; Bruulsema et al., 2008; Johnston and Bruulsema, 2014).

Right source means selecting the appropriate nutrient source which matches crop requirement and soil properties (Johnston and Bruulsema, 2014). Selection of proper fertilizer helps to ensure that appropriate nutrients are applied to crops to meet specific objectives and avoid unnecessary fertilization (Bryla, 2011). Selecting manure is quite challenging because different manures contain different amounts of nutrients (macro and micronutrients, and others) and organic matter depending on animal species, manure handling and management, bedding system, diets, and temperature (E. Gilley and M. Risse, 2000; Hernandez and Schmitt, 2012). Unlike commercial fertilizer, nutrients cannot be custom-blended. Also, estimating the availability of N in manure is more challenging than P or K estimation (Hernandez and Schmitt, 2012).

Estimating the right application rate of manure or fertilizer relies on knowledge of the previous crop, nutrients present in the soil, crop yield goal, nutrients present in manure or fertilizer, and nutrient availability (Franzen, 2010; Gerwig and Gelderman, 2005; Hernandez and Schmitt, 2012). Under application of N may decrease crop production, whereas over application can affect the environment (Johnston and Bruulsema, 2014). The appropriate application rate of N may fulfill crop N requirements and may also minimize N losses to atmosphere and water bodies (Bryla, 2011; Powers and Van Horn, 2001). However, N availability of crops and soil N losses vary with application methods, types of N sources, soil properties, and climatic factors.

Similarly, right timing for N application plays a crucial role in crop growth and to mitigate the possible N losses. Application of nutrient at the right time allows for adequate supply during crop demand (Bruulsema et al., 2008; Bryla, 2011; Johnston and Bruulsema, 2014). Also, N use efficiency may increase, and nitrate leaching reduces by applying a major part of the N in season, at or near the time when nitrogen demand is high (Charles et al., 2013). A study in Minnesota showed that application of fertilizer in spring increased N use efficiency by 20% compared to fall application and reduced nitrate loss by an average of 36% (Randall et al., 1992; Randall et al., 1997). Manure has some unique behaviors that are affected by the timing of application. Fall broadcasted, or injected manure allows more time to mineralize organic matters before crop uptake compared to spring application, however, more time available for potential soil N losses for fall application (Hernandez and Schmitt, 2012). The Maurer et al. (2017) study in

Iowa estimated the cumulative flux for fall injected swine manure was 3.48 k g ha^{-1} N_2O, whereas flux was 1.4 k g ha^{-1} N_2O for spring reapplication in a corn field.

Placement of fertilizer can influence nutrient uptake and crop yield (Reiman et al., 2008). Appropriate placement of fertilizer can help nutrient uptake, especially in soil which has a capacity of nutrient fixation (Johnston and Bruulsema, 2014). Soil inorganic distributions were altered by shallow and deep manure injections (Reiman et al., 2008).

Some of the other conservation practices are conservation tillage, growing cover crops, crop rotation, nitrification inhibitors, a slow-release fertilizer and amendment of organic matter. Conservation tillage or reduce tillage maintains the crop residues in the soil surface, increase infiltration, increase in soil organic carbon, enhance the soil quality, and improve soil resilience (Islam and Reeder, 2014; Uri, 2000). Also, conservation tillage as an effective practice in reducing N losses associated with soil erosion and surface runoff. Crop rotation practice may vary residual N available in soil (Sainju et al., 2017). Crop rotation practices may reduce the NO_3^- leaching from the agricultural lands (Zhu and Fox, 2003). The amount of reduction can be less, depending on the climatic condition and the rotation of crops (Randall et al., 1997). The rotation of legume and non-legume crops also shows a significant decrease in NO_3^--N loss (Randall et al., 1997). Cover crops are usually planted to reduce soil erosion, improve soil fertility and soil health (Sullivan and Andrews, 2012), improve water holding capacity and thus increases the effectiveness of N fertilizer applied in the field (U.S. Department of Agriculture, 2016), and reduce nutrient loss by leaching or in runoff (Baumhardt et al., 2015).

Nitrification inhibitors include chemicals added to the soils to stabilize fertilizer applied as NH_3 or in the NH_4^+ form by limiting the activity of the Nitroso-monas bacteria that convert NH_4^+ to NO_2^- (Dinnes et al., 2002). The nitrification inhibitors are used to slow the conversion process of applied NH_3 fertilizer, hold nitrogen in the field and reduce nitrogen losses before peak N demand for the crops. Use of nitrification inhibitors for N fertilizers rely on soil type and weather condition (Dinnes et al., 2002). Similarly, application of slow release fertilizer practice involves using less water-soluble materials to coat N fertilizers, which slows the entrance of water and slows down the dissolved N movement out of the coated area (Follett, 2008). Sulfur-coated urea is often used in agriculture fields as a slow-N release product (Follett, 2008).

2.4 Summary

Manure management is more challenging than inorganic fertilizer management due to variable nutrient concentration, bedding, and physical and chemical properties. Nitrogen availability from manure is a slow and gradual process which is affected by the type of manure, bedding materials, soil properties, weather condition, and management practices. Many conducted studies regarding nutrient availability, greenhouse gas emission, ammonia emission or nitrate leaching from the land application are focused on slurry or liquid manure application, though a significant amount of manure applied in the field is in solid form. Similarly, field studies related to solid cattle manure with and without bedding for N losses from cropland is limited. Thus, this study focused on fall-applied solid cattle manure with and without bedding on the corn-corn crop rotation field to understand the dynamics of N losses over fall and corn growing season, and to quantify the effect on the total soil nitrate, crop N, and yield.

CHAPTER 3 SOIL NITRATE, SOIL WATER NITRATE CONCENTRATION, CROP NITROGEN, AND YIELD FOR FALL APPLIED SOLID MANURE (WITH AND WITHOUT BEDDING) AND INORGANIC FERTILIZER FOR CORN PRODUCTION NEAR BROOKINGS, SOUTH DAKOTA

ABSTRACT: Nitrogen (N) is one of the major nutrients needed by all plants for their growth and reproduction. However, the excess losses of N from the soil not only decrease soil fertility and plant yield but can also impair water quality and air quality. The goal of this work was to understand the effect of fall-applied solid manure with bedding on nitrogen movement and transformations during corn production. The objectives of the research were to measure the soil nitrate (NO_3^--N), soil water nitrate (NO_3^--N) concentration, leaf and grain N, and yield from fall-applied N to a corn field, and compare the impact of different forms of applied N (solid beef cattle manure with bedding (MB), solid beef cattle manure only (MO), urea only (UO) and no-fertilizer (NF)), in Brookings County, SD over a two-year period. The application rate of plant-available N for manure and urea treatments were 130 kg ha^{-1} in Year 1 and 184 kg ha^{-1} in Year 2. The mean soil NO_3^--N was significantly higher for UO (105.3 kg ha^{-1}) compared to the other three treatments, whereas MB, MO, and NF were not significantly different with each other (71.7, 65.1, and 64.9 kg ha^{-1}). The Year, Growth Stage, and Year*Growth Stage interaction effects on total soil NO_3^--N were significant ($P < 0.05$). The total soil NO_3^--N at pre-planting stage for Year 2 was 18% greater than Year 1, whereas, at V6, it was decreased by 39% compared to Year 1. For soil water NO_3^--N concentration, Year 1 concentration (12.5 mg L^{-1}) was significantly greater than Year 2 concentration (6.5 mg L^{-1}). The average soil water NO_3^--N concentration between corn planting and vegetative

leaves six stage (V6) were not significantly influenced by any treatments. Leaf-N and grain-N concentrations tended to be different ($P < 0.1$) among treatments. The average leaf-N concentration for UO, MO, MB, and NF were 2.3, 2.1, 2.1, and 2.0%, respectively. Average grain-N concentrations were 1.4, 1.3, 1.2, and 1.2% for UO, MB, MO, and NF, respectively. The result showed that neither manure or urea treatments significantly affected yield compared to control from Year 1 data (Year 2 data was not available).

3.1 Introduction

Applying the correct amount of N at the correct time makes economic and environmental sense (Fageria and Baligar, 2005). Crops require a significant amount of N compared to other nutrients for growth and reproduction during the growth period. Manure (solid or liquid) can be applied to the soil to supply similar plant nutrients (N, P, K, and others) as commercial fertilizers, and application of manure can add organic matter in the soil and improve soil quality (Khaleel et al., 1981; Paul and Beauchamp, 1993; Van Faassen and Van Dijk, 1987). The majority of N in solid manure is in organic form, so, the release of plant usable N depends on mineralization. Thus, the application rate and timing of manure application should be determined based on N-releasing capacity (Qian and Schoenau, 2002). The estimated rate of manure based on its N-release capacity may supply necessary quantity of N to the crops and may reduce the possibility of an excess amount of N loss via leaching under the root zone (Qian and Schoenau, 2002). Nitrogen release capacity from manure may vary with rate and timing of manure application, and without addressing these factors may result in insufficient N to crops or harmful losses to the environment (Smith and Chambers, 1993; Sutton, 1994). Nitrogen loss via leaching from agricultural land can pollute the groundwater and surface water and may create a problem for aquatic ecosystem due to toxic algal blooms and lack of oxygen (Carpenter et al., 1998; Dinnes et al., 2002). Thus, proper manure management practices play a vital role in enhancing crop production and for decreasing environmental impacts (Fageria and Baligar, 2005; Jokela, 1992). Other loss mechanisms are runoff and volatilization (Bierman and Rosen, 2005; Lamb et al., 2014b; Paramasivam et al., 2009; Provin and Hossner, 2001).

Loecke et al. (2004) conducted a study about the effect of fresh and composted solid swine manure and time of manure application (fall or spring) on nutrient status and yield in a corn-field. They found no significant difference in corn yield due to the form of solid manure and time of application in 2000, whereas in 2001, corn yield for fall application (composted manure) was higher than spring application (fresh manure). Also, their result showed the average N supply efficiency was highest for fall-applied composted manure (34.7%) compared to fall-applied fresh manure (24.3%), and spring applied composted manure (25%) while applying the same amount of manure (340 kg total N ha^{-1}). However, application of manure in the fall can be a high risk for NO_3^--N leaching, mainly in sandy soils (Van Es et al., 2006). Another study reported the time of manure application had little or no effect on yield response (Jokela, 1992). Ndayegamiye and Cote (1989) found that soil organic carbon, microbial activities, and potentially mineralized-N were positively correlated with the application rate of farmyard manure or pig slurry.

Many studies have been conducted to show or compare the effect of solid and liquid manure (mainly poultry and swine manure) on soil NO_3^--N or soil water NO_3^--N leaching and corn yield. However, limited studies have quantified the effect of solid beef cattle manure with or without bedding on soil nitrate concentration, soil water nitrate concentration, and yield in the corn-field in Northern Great Plain area. This study focuses on how solid beef feedlot manure with bedding (corn Stover) can influence total soil nitrate, leachate nitrate concentration, leaf-N, grain-N, and corn yield compared to only solid beef manure or urea application. The objectives of the research were to measure the soil NO_3^--N, soil water NO_3^--N concentration, leaf and grain N, and yield from fall-applied N to a corn field, and compare the impact of different forms of applied N (solid beef cattle manure with bedding (MB), solid beef cattle manure only (MO), urea only (UO) and no-fertilizer (NF)), in Brookings County, SD over a two year period.

3.2 Materials and methods

3.2.1 *Sites description*

The research site (South Dakota Felt Farm) was located near Brookings County (44º 22’ 07.5” N and 96º 47’ 35.7” W, and 516 m above mean sea level) and was established in the fall of 2015. The research site area was 0.11 ha (150 ft x 81 ft) with an average slope less than 1%. The soil was categorized as a silty clay loam soil and classified as a Udic Haploborolls (Schaefer, 2005). The 2015 crop was soybeans. The corn planting date was May 2 in 2016. In 2017, corn was first planted on May 6 and replanted on June 2 following a lack of sufficient seed emergence, attributed to weather and field conditions. Daily precipitation values for the research site were obtained from the South Dakota Climate and Weather Station.

3.2.2 *Treatment and experimental setup*

The treatment design was a randomized complete block design with plots as the experiment unit (3.3 m x 9.1 m). The four treatments, solid beef cattle manure with bedding (MB), beef cattle manure without bedding (MO), urea (UO), and no fertilizer/control (NF), in each Block (4 Blocks) were assigned randomly. Prior to laying out the plots in 2015, we collected soil samples (0-60 cm) randomly in the research field, and the average total soil NO_3^--N was 112 kg ha^{-1}. In 2016, prior to N application the average total soil NO_3^--N was 58 kg ha^{-1}.

Based on the soil test, manure tests, and yield goal, nitrogen-based application rates for manure with or without bedding and urea fertilizer were determined using the South Dakota Fertilizer Recommendations Guide EC-750 (SDSU, 2005) (Table 3.1). The corn yield goal was 1.13 kg m^{-2} (180 bushels $acre^{-1}$). Manure with and without bedding and urea were applied on November 3 in 2015, and November 16 in 2016. After manual application of manure and urea on the plots, the N-source was incorporated within 24 h through two passes with a disk plow.

Table 3.1 Application rate and physical characteristics of manure and urea fertilizers for experiment site at South Dakota Felt Farm, Brookings, South Dakota

Variable	Year	Treatment[z]		
		MB	MO	UO
Application Rate (kg ha^{-1})	2015	32505	33850	283
	2016	37661	29590	400
Moisture content (%)	2015	74.2	72.3	-
	2016	69.1	53.0	-
Total N (g kg^{-1})	2015	8.5	8.2	460
	2016	8.5	11.5	460
Ammonium-N (g kg^{-1})	2015	1.85	1.77	-
	2016	1.62	1.16	-
Dry matter (%)	2015	25.8	27.7	-
	2016	31.0	47.0	-

[z] MB = solid beef cattle manure with bedding; MO = solid beef cattle manure only, UO = urea only

3.2.3 *Sample collection and analysis*

The soil sampling frequency was related to the N management and crop growth stages: before manure application, before planting, six leaves vegetative stage (V6), and postharvest stage from each plot. Each sampling day, a total of 32 composite soil samples were collected at 0-15 cm (0-6 in.) and 15-60 cm (6-24 in.) depths in each plot using a probe auger. The shallow soil samples (0-15 cm) were analyzed for NH_4^+-N, electrical conductivity (EC), organic matter (OM), phosphorus (P) concentration, total N, total C, and pH. The NO_3^--N for 0-15 and 15-60 cm depths were measured in each sample and added together to get total soil NO_3^--N. All the samples were analyzed by AgLabExpress, Sioux Falls. The pH, EC, and OM were analyzed using North Central Extension Research Activities guidelines (NCERA-13, 2015). Soil nitrate was analyzed using Lachat Nitrate method with Bray extraction. Similarly, Olsen P, ammonium, and Total N and C in the soil samples were analyzed using Lachat Phosphorus, Lachat Ammonia, and Dumas method, respectively.

Soil surface (0-5 cm) samples were collected from each plot using the AMS bulk density soil sampling mini kit (with 5 cm * 5 cm stainless steel ring) for determining the bulk density of soil. Soil bulk density was determined using Aridlands Ecology Lab Protocol (modified 2009.01.19, S. Castle). In this protocol, the collected soil samples were oven dried at 105°C for 48 hours and weighed. The bulk density was calculated by dividing the dried weight of the soil by volume of the ring.

One suction lysimeter (127 cm in length and 2.2 cm diameter; Irrometer Company, Inc., CA, USA) was installed in each plot at 120-cm soil depth in the north end of each plot (Figure 1). Soil water samples were collected on days 17, 23, 31, 35, 44, and 50 after planting in Year 1, and on days 16, 24, 33, 39, 48, and 53 after day of planting in Year 2. The number of soil water sampling days depended on rainfall events and soil water availability. During sample collection, a hand pump applied a vacuum pressure between -60 to -70 kPa and the vacuum was maintained for 4 hours. Soil water collected in the lysimeters was extracted using a polypropylene syringe, collected into a polypropylene vial, and transferred to the laboratory for analyses. In the laboratory, NO_3^--N concentration in collected water sample was determined using and Automated Timberline TL2800 Ammonia Analyzer (Timberline Instruments, Boulder, CO).

Leaf samples were collected from each plot at the six leaves (V6), tasseling (VT), and physiological maturity (R6) stages for leaf nutrient analysis (total N percentage). The six most recently unfurled leaves below the whorl at the V6 stage, and six leaves below the corn ear at VT and R6 stages were collected from six plants in each plot and composited. Plant samples were dried in paper bags at 65-70°C in forced-air dryer. They were then ground to pass an 18-mess (1 mm opening) stainless steel screen with Wiley mill. The samples were then stored in paper envelopes. They were dried overnight at 65-70°C just prior to analysis. Total nitrogen (in %) was measured using the micro-Kjeldahl procedure by the South Dakota State University Soil Testing and Plant Analysis Lab, Brookings. In the micro-Kjeldahl method, a 0.4 ± 0.01 g sample was weighed, digested and then distilled. Then, the N% was calculated using Equation 3.1:

$$\% \text{ N} = \frac{(\text{ml of acid} - \text{blank}) * (\text{normality of acid}) * 0.014 * 100}{\text{weight of sample}} \tag{3.1}$$

Yield and corn-grain samples were collected during the Year 1 (2016) harvest. The N concentration in the corn grain was analyzed using inductively coupled plasma (ICP) analysis by the South Dakota State University Soil Testing and Plant Analysis Lab, Brookings. Yield and grain samples were not collected during the 2017 harvest.

3.2.4 *Statistical analysis*

The experimental design was a randomized complete block design and was analyzed as a mixed-effect model. The data for soil NO_3^--N water concentration, leaf-N concentration, and total soil NO_3^--N were repeated measurements on the plots (experimental units). Treatment (MB, MO, UO, and NF) and Growth Stage (stages differed for the various dependent variables) were considered fixed factors for total soil

NO_3^--N and leaf-N concentration data, but Time was considered as a random factor for soil water NO_3^--N data. The Year (Year 1 and Year 2) was considerd a fixed effect and Block (replication) considered as a random factor for all variables. All the data analyses (soil NO_3^--N water concentration, leaf-N concentration, total soil NO_3^--N, grain-N, and yield) were performed in SAS using the PROC GLIMMIX procedure (SAS-Institute, 2012). The mixed model approach included the effects of N source or Treatment, Growth Stage/Time, Block, Year, and their interactions between these variables. Significant differences were considered at $P < 0.05$. The normality of the residuals was reviewed using Q-Q plots and if residuals appeared not normal, different distribution options (e.g., lognormal, exponential, Poisson) available in PROC GLIMMIX were tested. Different covariance structures were used to assess the repeated measure data, including covariance component (VC), compound symmetry (CS), auto-regression (AR (1)), unstructured (UN), and Toeplitz (TOEP). The covariance structure selected for each variable was based on the smallest Akaike information criterion (AIC) value.

Normal distribution and AR(1) were used for soil water NO_3^--N concentration and leaf-N, whereas normal distribution and VC were selected for grain-N and yield data. However, lognormal distribution and UN covariance structure were selected for total soil NO_3^--N dataset. The least square means (lsmean) from lognormal distributions were back transformed for reporting purposes. For post hoc test, Tukey’s Honest Significant Difference (HSD) was used. A correlation analysis was done using yield and leaf-N concentration data from each plot (16 plots) at VT stage (number of samples, N = 16), and the annual average total soil nitrate for Year 1 only using PROC CORR.

3.3 Results and discussions

3.3.1 *Total soil nitrate*

Table 3.2 presents the average soil NO_3^--N levels among Blocks for the Treatments, Years and Growth Stages.

Table 3.2 Mean (±SE[y]) of total soil nitrate for four Treatments with Year by Growth Stage

Year	Growth Stage	Treatment[z]				Mean
		MB	MO	UO	NF	
Year 1	Pre-plant	153.3±19.5	155.1±19.8	304.1±38.7	151.9±19.4	181.2±14.5
	V6	67.3±16.6	50.2±12.4	109.7±27.1	57.5±14.2	66.5±8.8
	Post-Harvest	57.3±7.5	44.5±5.9	63.7±8.4	34.8±4.6	48.5±4.0
Year 2	Pre-plant	182.7±23.3	172.2±21.9	422.3±53.8	159.7±20.3	213.6±17.1
	V6	39.2±9.7	38.7±9.7	43.4±10.7	44.7±11.1	40.6±5.3
	Post-Harvest	34.3±4.5	35.2 ± 4.6	37.5±4.9	38.8±4.8	35.7±2.9
Overall mean		71.7±6.3[b]	65.1±5.7 [b]	105.3±9.2[a]	64.9±5.7[b]	

[y] Mean (±SE) = Estimate mean (±Standard Error) obtained from Year * Treatment Least Squared mean table; Superscript of different letters within treatments indicate significantly different ($P < 0.05$).

[z] MB = solid beef cattle manure with bedding; MO = solid beef cattle manure only, UO = urea only; NF = no-fertilizer.

The mean total soil NO_3^--N for the UO plots was significantly higher than manure and no fertilizer plots ($P < 0.05$, Figure 3.1). The lower soil NO_3^--N in the manure-treated plots may be related to slow mineralization of manure organic matter, particularly in cold weather, and low inorganic N in manure compared to urea. Carbon and organic matter can reduce N mineralization in manure plots. Qian and Schoenau (2002) found that N mineralization decreases significantly with increase C/N ratio in cattle manure.

Figure 3.1 Mean total soil nitrate and standard error (vertical lines) for four treatments over corn growing season. (MB, MO, UO, and NF are solid beef cattle manure with bedding, solid beef cattle manure only, urea only, and no-fertilizer treatments, respectively, whereas different letters above the bars indicate significant differences ($P < 0.05$))

The interaction between Year and Growth Stage was also a significant effect on total soil nitrate ($P < 0.05$, Figure 3.2). The total soil NO_3^--N in Year 2 at the pre-plant stage was about 18% greater than Year 1. However, total soil NO_3^--N was significantly lower at the V6 stage by 39% and at the post-harvest stage by 26% in Year 2 compared to Year 1 (Table 3.2 and Figure 3.2). The significant difference of total soil NO_3^--N between the two years at the various corn stages might be due to the replanting of the crop in Year 2 (1 month after the first plant); and some excess quantity of NO_3^--N could be lost via leaching, volatilization or denitrification compared to the shorter period prior to V6 in Year 1.

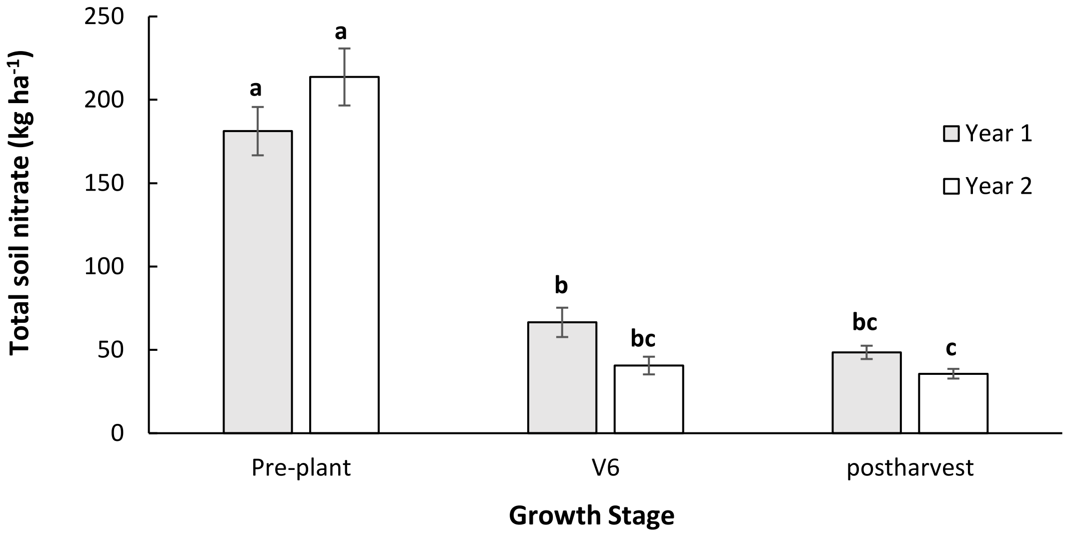

Figure 3.2 Mean total soil nitrate with standard error (vertical lines) for Year by Growth Stage. (Different letters above the bars indicate significant differences ($P < 0.05$))

3.3.2 *Soil water nitrate concentration*

Soil water samples from each plot were collected between corn planting and the V6 stage in both corn growing seasons (Year 1 and Year 2) to determine leachate NO_3^--N concentration. Treatments were not significantly different ($P > 0.05$; Table 3.3), which indicates that short-term leachate NO_3^--N concentrations from the soil profile (1.2 m depth) were not significantly affected by manures or fertilizer application. In several instances and on average for the MO and UO treatments, the mean concentration of soil water NO_3^--N for MO and UO were about 13.8 and 10.3 mg L^{-1}, respectively. The U.S.

Environmental Protection Agency standard (>10 mg NO_3^--N L^{-1}) for drinking water is 10 mg L^{-1} (EPA, 2002).

Table 3.3 Mean (±SE[y]) of soil water leachate nitrate (NO_3^--N) concentration for four Treatments for Year 1 and Year 2

Year	Treatment[z]				Mean
	MB	**MO**	**UO**	**NF**	
Year 1	9.0 ± 3.2	16.6 ± 3.2	12.1 ± 3.2	12.4 ± 3.2	12.53 ± 3.2^a
Year 2	3.7 ± 3.4	9.1 ± 3.3	8.5 ± 3.4	4.5 ± 3.3	6.45 ± 3.4^b
Mean	6.35 ± 3.3^a	12.85 ± 3.3^a	10.3 ± 3.3^a	8.45 ± 3.3^a	9.49 ± 3.3

[y] Mean (±SE) = Estimate mean (±Standard Error) obtained from Year * Treatment Least Squared mean table; Superscripts of same letters within Treatments indicates not significant difference ($P < 0.05$).

[z] MB = solid beef cattle manure with bedding; MO = solid beef cattle manure only; UO = urea only; NF = no-fertilizer

The average soil water NO_3^--N concentration was significantly greater in Year 1 compared to Year 2. The possible reasons for the significant difference between two years are variations in total soil NO_3^--N and rainfall. The total soil NO_3^--N in Year 2 was initially higher, but lower at the V6 stage compared to Year 1 (Figure 3.2). A potentially lower soil NO_3^--N level in Year 2 during the soil water NO_3^--N sample collection was possible with re-planting in Year 2 and the month delay in the calendar year, relative to Year 1. However, rainfall was also another factor that may have influenced soil water NO_3^--N concentration. The rainfall in Year 1 between planting and V6 stage was about 148 mm, which was greater than the 108 mm of rainfall in Year 2 during the same period of corn growth. Allaire-Leung et al. (2001) found that nitrate leaching measured at 1-m depth by ion-exchange resign bags was positively correlated to soil NO_3^--N. Nitrate leaching from soil also depends on soil type, N application rate, types of N sources, cover crops cropping intensity and crop N uptake (Aronsson and Bergström, 2001; Havlin et al., 2005; Wyland et al., 1996), and these factors influence translation of these research results to other fields and crop systems.

3.3.3 *Leaf nitrogen concentration*

The average leaf-N concentration was significantly affected by the Year, Growth Stage (V6, VT, and R6), and the interaction between Year and Growth Stage ($P < 0.05$, Table 3.4 and Figure 3.3). The mean leaf-N tended to be different between treatments ($P < 0.1$).

Table 3.4 Mean (SE[y] = 0.14) leaf N% for four Treatments for Year 1 and Year 2

Year	Growth Stage	Treatment[z]				Mean
		MB	MO	UO	NF	
Year 1	V6	2.93	2.99	3.13	2.77	2.96±0.08
	VT	2.79	2.72	2.90	2.84	2.81±0.08
	R6	1.50	1.45	1.45	1.26	1.42±0.08
Mean (Year 1)		2.41±0.09	2.39±0.09	2.50±0.09	2.29±0.09	**2.39±0.05[a]**
Year 2	V6	2.85	2.73	3.10	2.64	2.83±0.08
	VT	1.70	1.69	2.06	1.56	1.75±0.08
	R6	1.06	1.08	1.03	1.04	1.05±0.08
Mean (Year 2)		1.87±0.09	1.83±0.09	2.06±0.09	1.75±0.09	**1.88±0.05[b]**
Overall mean		2.14±0.07	2.11±0.07	2.28±0.07	2.02±0.07	

[a] Mean (±SE) = Estimate mean (±Standard Error) obtained from Year * Growth Stage * Treatment Least Squared mean table. Superscripts of different letters within years indicates significant difference ($P < 0.05$).

[z]MB= solid beef cattle manure with bedding; MO= solid beef cattle manure only; UO =urea only; NF =no-fertilizer.

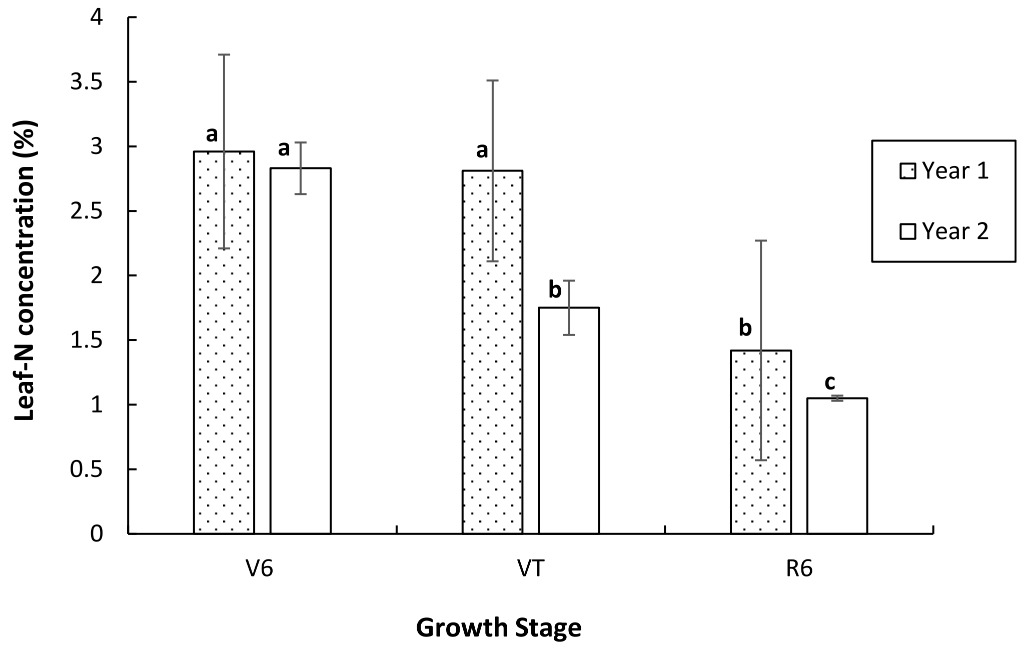

Figure 3.3 Mean leaf-N concentraton with standard error (vertical lines) for Year 1 and Year 2 based on Growth Stage. (V6, VT, and R6 are six-leaves stage, taseling stage, and maturity stage of corn, respectively; different letters above the bars indicate significant differences (P < 0.05))

The mean leaf-N concentration for UO was higher compared to remaining treatments (Table 3.4). The variation in leaf-N concentration may be related to differences in soil NO_3^--N between treatments. Furthermore, the result showed the leaf-N concentration at V6 stage was higher in both years of study and was decreasing over growth stage (Figure 3.3). The average leaf-N was 2.4% for the Year 1, whereas for Year 2, it was 1.9%. The significant variation in leaf N concentration among two years may be due to a variety of corn, rainfall, soil moisture, and soil available N late corn planting in the Year 2.

3.3.4 *Grain nitrogen and corn yield*

Grain N and yield were only collected and analyzed in Year 1.

The mean grain-N tended to be different among treatments ($P < 0.1$). In Year 1, the mean grain-N for UO was 1.36%, whereas concentration of grain-N for MB, MO, and NF were 1.33, 1.22, and 1.19%, respectively (Table 3.5).

The average corn yield was not statistically different between treatments. The average yield from UO, MB, MO, and NF treatments were about 1.25, 1.20, 1.08, and 1.08 kg m^{-2} (Table 3.6), respectively.

Table 3.5 Mean (±SE)[y] grain-N and corn yield for four Treatments for Year 1

Variable	**Treatment[z]**				**Mean**
	MB	**MO**	**NF**	**UO**	
Grain N (%)	1.33 ± 0.05	1.22 ± 0.05	1.19 ± 0.05	1.36 ± 0.05	1.28
Yield (kg m^{-2})	1.20 ± 0.08	1.08 ± 0.08	1.08 ± 0.08	1.25 ± 0.08	1.15

[y] Mean (±SE) = Estimate mean (±Standard Error) obtained from Treatment Least Squared mean table.
[z]MB= solid beef cattle manure with bedding; MO = solid beef cattle manure only; UO =urea only; NF =no-fertilizer.

The Year 1 correlation results showed that leaf-N at VT stage and yield were significantly related ($r = 0.70$, and $P < 0.05$). Kovács and Vyn (2017) found ear-leaf N concentration at mid-silking stage and corn yield significantly correlated. Similarly, Voss et al. (1970) showed corn yield was positively related with leaf-N concentration. The yield and grain-N also varied significantly with the amount of total NO_3^--N present in the soil. For Year 1 (data, n=16 for grain-N and yield), grain-N and yield were positively correlated with average total soil NO_3^--N ($r = 0.71$ and $r = 0.68$).

3.4 Conclusions

The total soil NO_3^--N was significantly affected by fall-applied manure and fertilizer treatments for the two-year experiment between the Fall of 2015 and Fall of 2017 in silty clay loam soil in corn production near Brookings, SD. Our experiment showed the average of total soil NO_3^--N for the UO treatment was significantly greater than manure treatments or no fertilizer over the two corn production years compared to manure treatments, however, they received the same amount of plant-available N based on crop yield. The result also showed the Year and Growth Stage interaction significantly affected total soil NO_3^--N. The significant effect of Year*Growth Stage interaction on total soil NO_3^--N could be associated with replanting because replanting delayed about 1-month compared to Year 1. During that 1-month period, soil could lose N via leaching, volatilization, and denitrification. During this period, the manure and urea treatments did not significantly differ in effects on soil water NO_3^--N concentration, even compared to no-fertilizer. In contrast, soil water NO_3^--N was significantly higher in Year 1 (12.5 mg L^{-1}) compared to Year 2 (6.5 mg L^{-1}). A significant change in soil water NO_3^--N between two years could be due to rainfall or significant change in total soil NO_3^--N. The leaf-N and grain-N tended to be different among the manure and urea treatments, with the UO treatment producing the highest N concentration, and NF resulting in the lowest concentration. We found the corn yield was not significantly different among any treatments (manure, urea or no fertilizer) in Year 1 (Year 2 measurements were not collected). Overall, we observed that the total soil NO_3^-- N was greatly influenced by urea compared to solid beef cattle manure with and without bedding, but the effect on yield was non-existent in Year 1, and leaf-N and soil water NO_3-N in Year 1 and Year 2.

CHAPTER 4 AMMONIA AND NITROUS OXIDE FLUXES FOR FALL APPLIED SOLID MANURE (WITH AND WITHOUT BEDDING) AND INORGANIC FERTILIZER FOR CORN PRODUCTION NEAR BROOKINGS, SOUTH DAKOTA

ABSTRACT: Land applied manure or fertilizer can contribute to soil N losses in the form of ammonia (NH_3) and nitrous oxide (N_2O) gases. Both gases are responsible for air quality deterioration. The goal of this work was to understand the effect of fall-applied solid manure with bedding on nitrogen movement and transformations during corn production. This study focused on comparing the effects of fall-applied solid beef cattle manure with and without bedding (MB and MO), urea (UO) and no-fertilizer (NF) on NH_3 and N_2O fluxes from silty clay loam soil under a corn-corn cropping system, near Brookings, SD. The methods for collecting samples for soil N fluxes were semi-static open chambers for NH_3 and static chambers for N_2O. The results showed the average NH_3 flux for MB (3.4 ± 0.9 g ha^{-1} h^{-1}) was significantly higher compared to NF, whereas MO and UO were not significantly different than MB and NF treatments. The aerial N_2O flux released from UO (79.0 ± 24.9 µg m^{-2} h^{-1}) was significantly greater than NF, though the N_2O fluxes from manure treatments were not significantly different compared to UO and NF. The NH_3 and N_2O fluxes for NF were 1.4 ± 0.4 g ha^{-1} h^{-1} and 24.6 ± 7.7 µg m^{-2} h^{-1}. Understanding soil N loss paths and related factors will help to identify appropriate management practices and nutrient management plans to mitigate excessive N losses to the environment.

4.1 Introduction

Nitrogen (N) is one of the most essential nutrients for corn grain production (Havlin et al., 2005a; Provin and Hossner, 2001; Robertson, 1997). However, N loss may create environmental problems resulting in the decrease of soil, water, and air quality (Cameron et al., 2013; Mosier et al., 2004). The risk of N loss from agricultural land to the atmosphere increases with excess use of N sources and their mismanagement (Dinnes et al., 2002; Provin and Hossner, 2001) and gaseous losses are mainly NH_3 and N_2O, which produce air quality issues (Paramasivam et al., 2009; Rotz et al., 2014; Wu et al., 2008). Nitrous oxide has a global warming potential which tends to be about 310 times higher than that of a carbon dioxide (CO_2) molecule and contributes to depletion of the ozone layer (Chapuis-Lardy et al., 2007; IPCC, 2014; Robertson et al., 2000; Schlesinger, 2009). Ammonia release and deposition into the atmosphere can lead to acidification and eutrophication (Huijsmans et al., 2003; Nielsen et al., 2003). Also, emission of NH_3 to the atmosphere plays a role in the formation of airborne fine particulate matter by reacting with sulfur dioxide and oxides of nitrogen (Behera and Sharma, 2010; Bittman et al., 2014; Gong et al., 2013). The fine particulate matter can responsible for adverse health effects (Dabek-Zlotorzynska et al., 2011; Pope III and Dockery, 2006; Pope III et al., 2009). The result of soil N loss reduces crop N uptake (Bouwman et al., 2002; Paramasivam et al., 2009), and therfore impacts crop production and economics (Cassman et al., 2002; Fageria and Baligar, 2005).

The release of NH_3 and N_2O gases from agriculture into the atmosphere has been increasing due to human activities. EPA (2016) indicated that agricultural activities such as manure and fertilizer application, cropping practices are significant sources which are responsible for about 79% of total US N_2O emissions. Emission of N_2O varied from year to year, however, between 1990 and 2014, overall N_2O emissions increased by 5.9 percent (EPA, 2016). Ammonia volatilization is one of the primary loss mechanisms of N from agricultural production systems (Bouwman et al., 2002; Smil, 1999). Emission of NH_3 from agriculture contributes 20 to 80% of applied manure TAN, while animal manure and N fertilizer are the major contributors (Aneja et al., 2008; Misselbrook et al., 2000). However, Jantalia et al. (2012) found the range of NH_3-N loss was between 0.1 and 4% of total N from surface-applied N fertilizers.

Many factors influence the N_2O and NH_3 flux and emission from land-applied manure and fertilizers. Manure and fertilizer types, their characteristics, application rate, methods, and timing and some other management practices like reduced tillage, crop rotations, and cover crops, soil properties, climatic condition are the primary controlling factors for soil surface emission (Cai et al., 2016; Engel et al., 2010; Meisinger and Jokela, 2000; Miola et al., 2014; Paramasivam et al., 2009). Also, bedding materials and their types can affect manure characteristics which may impact surface emissions (Miller et al., 2012; Misselbrook and Powell, 2005). Miller et al. (2012) researched loss of N by denitrification due to long-term application of composted (with straw bedding) versus fresh feedlot beef cattle manure. The studied showed a significantly lower cumulative denitrification flux for composted manure (with straw bedding) (0.7-1.4 kg N_2O-N ha^{-1}) compared to fresh feedlot manure (3.2-5.1 kg N_2O-N ha^{-1}). Paramasivam et al. (2009) found cumulative NH_3 volatilization loss over 19 days from poultry litter manure and swine manure were 4 to 27% and 14 to 32% of applied total N, respectively.

Several studies have compared the effects of slurry or liquid manure application of different manure types (swine, dairy, poultry, feedlot) on N_2O and NH_3 emission from the soil surface (Agnew et al., 2010; Amon et al., 2006; Beauchamp et al., 1982; Dustan, 2002; Gordon et al., 2001; Jarecki et al., 2008; Meisinger and Jokela, 2000; Miola et al., 2014; Rochette et al., 2001). However, limited field studies have been conducted to measure N_2O and NH_3 soil surface flux with solid beef cattle manure (with and without bedding) applied. Understanding the influence of bedding on N losses in gaseous form from surface-applied solid manure can help refine management and nitrogen loss factors.

The research aimed to measure the influence of fall-applied solid beef manure, with or without bedding, and urea application on NH_3 and N_2O fluxes during corn production and compare these fluxes no manure/fertilizer N application.

4.2 Materials and methods

4.2.1 *Site description*

The research site was established in the fall of 2015 at the South Dakota State University Felt Research Farm near Brookings (44º 22’ 07.5” N and 96º 47’ 35.7” W, and 516 m above mean sea level). The area covered by the research field was about 0.11 ha (150 ft x 81 ft) with an average slope less than 1%. The soil was silty clay loam soil and classified as a Udic Haploborolls (Schaefer, 2005). The 2015 crop was soybeans. The corn was planted on May 2 in 2016. In 2017, the corn was first planted on May 6, and replanted on June 2 following a lack of sufficient seed emergence, attributed to weather and field conditions.

4.2.2 *Treatment and experimental setup*

The treatments (plots) were randomly assigned within blocks (Figure 4.1) and plots were considered the experiment unit (3.3 m x 9.1 m). The four treatments in each Block were: solid beef cattle manure with bedding (MB); solid beef cattle manure without bedding (MO); urea (UO); and no-fertilizer/control (NF). There were four replicates of each treatment. Manures with and without bedding and urea were applied on November 3 in 2015 and November 16 in 2016. Nitrogen application rates were determined prior to application based on soil and manure analyses and yield goal, using the South Dakota Fertilizer Recommendations Guide EC-750 (SDSU, 2005). The corn yield goal was 1.13 kg m^{-2} (180 bushels/acre).

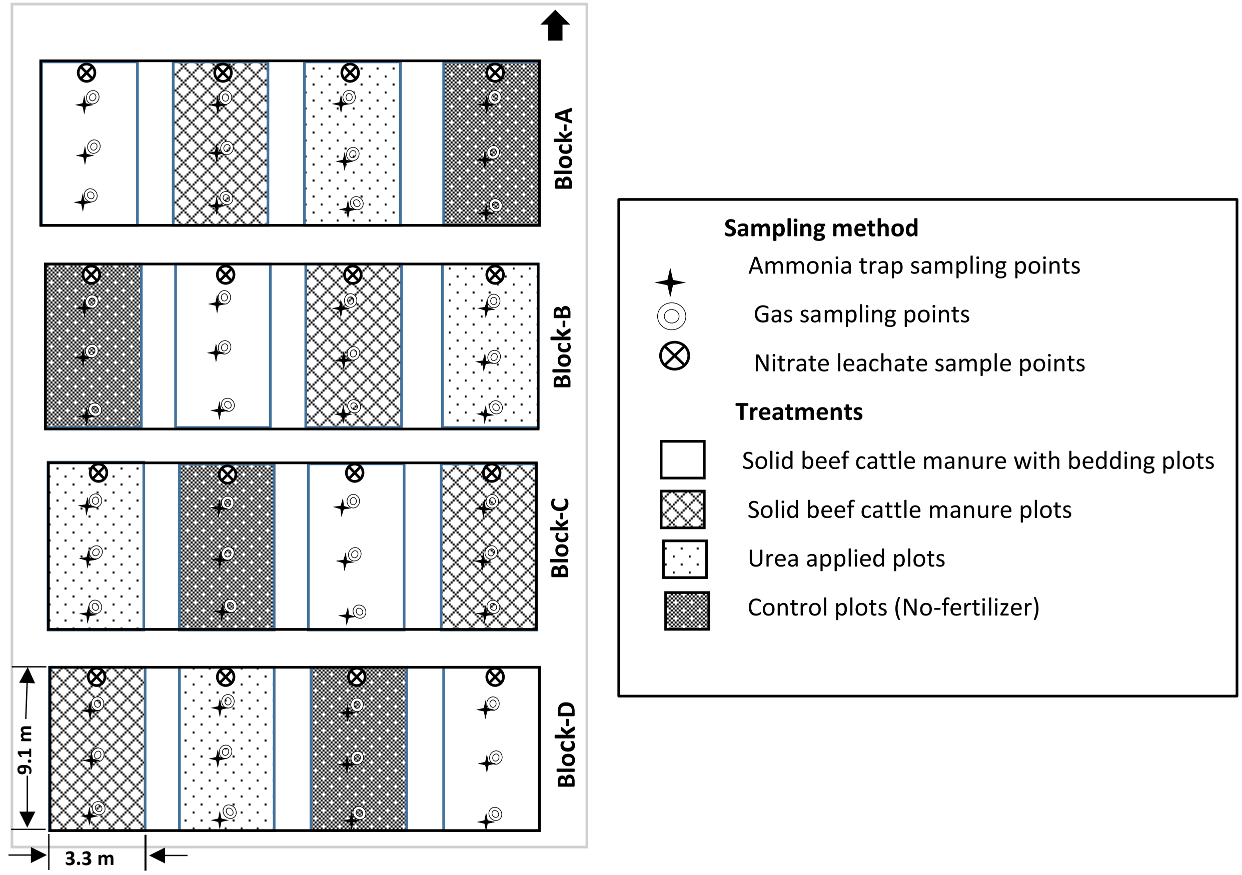

Figure 4.1 Layout of the experimental site at the South Dakota State University Felt Farm (Brookings County).

The application rates of beef feedlot manure with bedding were 3.25 kg m^{-2} and 3.77 kg m^{-2} in the Fall of 2015 (Year 1) and 2016 (Year 2), respectively. The application rates for solid beef manure (no bedding) were 3.39 kg m^{-2} in Year 1 and 2.96 kg m^{-2} in Year 2. Urea was applied at rates of 28.3 and 40.0 g m^{-2} in Year 1 and Year 2, respectively. Manure and urea were applied manually to the plots and incorporated within 24 h through two passes with a disk plow. Manure was from a beef feedlot and the bedding (where applicable) was corn Stover. The manure characteristics are described in detail in Chapter 3; however, it is worthwhile to note the ammonium-N concentrations were 1.85 and 1.70 g kg^{-1} for MB and MO, respectively, in Year 1; and 1.62 and 1.16 g kg^{-1} for MB and MO, respectively, in Year 2.

4.2.3 *Sample collection and analysis*

Composite samples of ammonia (NH_3) gas release were collected in three locations of each plot using semi-static chambers and with acidified foam strips as described by Jantalia et al. (2012). In Year 1, the samples were taken on days -4, 3, 7, and 13 d from the day of N application in the fall, and -6, 10, and 30 d from the day of planting in 2016. In Year 2, the sampling days were -7, 1, 6, and 15 d from the day of N application in 2016, and -35, 7, and 42 d from the day of replanting in Year 2. On the day of measurement, the foam strips and acid solution were collected, stored in a medium sized plastic freezer bag, and new traps (acidified strips) were inserted. The collected ammonia sample traps were transferred to the laboratory and kept in the freezer. In the laboratory, the thawed samples traps were extracted with 250 mL of 2 M KCL solution. Forty ml of this solution were then sealed and frozen at –18 °C in polypropylene vials before analysis. Samples were analyzed using an Automated Timberline TL2800 Ammonia Analyzer (Timberline Instruments, Boulder, CO).

Ammonia concentration was obtained in g N ha^{-1} by multiplying NH_3 concentration (µg mL^{-1}) and the total volume of solution (250 mL), then dividing by the surface area of the soil covered by the respective chamber (79 cm^2). The ammonia flux (g N ha^{-1} h^{-1}) for each plot was determined by dividing ammonia concentration by elapsed time from installation to the removal of the NH_3 traps.

For nitrous oxide gas (N_2O) sampling, three polyvinyl chloride (PVC) collars (25.4 cm internal diameter by 15 cm height) extending 5 cm above the soil surface were installed on each treatment plot (n=48). Gas samples were collected on days -4, 7, and 13 in the Fall of Year 1, and -7, 1, 6, and 15 in the Fall of Year 2 from the day of manure/urea application. Also, gas samples were collected on days 7 and subsequent monthly intervals following corn planting to August in both experimental years, preferably after a rainfall event. In Year 1, pre-planting N_2O samples were collected on days -8 from the day of corn planting, whereas, in Year 2, it was collected on days -38 and -17 from replanting. On each sampling day, the vented PVC chamber caps (5872 cm^3) were placed on the collar, and gas samples were withdrawn from each chamber after 0, 30 and 60-minutes following the static chamber method, described by Parkin and Venterea (2010). During each withdrawal, 10 ml gas samples were drawn using a 30-ml syringe and transferred to 12 ml pre-evacuated glass vials sealed with butyl rubber septa. Also, one sample of ambient air was collected during the sampling time for each Block to measure the concentration of nitrous oxide in the atmosphere. All the samples (0, 30 and 60 min) including ambient air sampling were collected between 9:30 am to 4:00 pm. These collected gas samples were analyzed for N_2O concentrations using a Gas Chromatograph (Shimadzu 14B with a CombiPal AOC-5000 auto-sampler, a flame ionization detector [FID] and an electron capture detector [ECD], Shimadzu Corporation, Japan).

Ancillary parameters including air and soil temperature (Acurite Digital Meat Thermometer, 00641W, AcuRiteNSF) and soil moisture (HH2 Moisture Meter, Theta probe type ML2x, Delta-T Devices Cambridge, England) were collected around each chamber on each gas sampling day.

Based on review of the 0 min and ambient concentration samples, the average ambient concentration measurement was used in place of the 0-min samples for each sampling day. The N_2O flux (µL N_2O L^{-1} h^{-1}) were determined from N_2O concentrations relative to elapsed time. Flux calculations were not performed if: (a) the time 30-min (T30) and/or time 60-min (T60) concentration(s) were less than (1- error)*ambient concentration; (b) the quadratic curve through the 3 data points was concave down and T60*(1+error) was less than T30*(1-error); or (c) the quadratic curve through the 3 data points was concave up and a linear slope fit through the 3 points was not significantly different than zero. If the quadratic curve through the 3 points was concave down, the first order coefficient of the quadratic equation fit through the 3 data points was considered the flux. If the quadraric curve through the 3 points was concave up, but the linear slope through the 3 points was significantly different than zero, the slope was considered the flux. The allowable error (proportional to concentration) was 20%. Evaluated N_2O fluxes were then converted into µg N_2O -N m^{-2} h^{-1} using the Ideal Gas Law equation. The resulted fluxes were corrected using soil properties (bulk density, clay fraction, pH, moisture content, and soil temperature) using method derived from Venterea (2013).

4.2.4 *Weather data*

Daily precipitation and maximum and minimum mean air temperatures from the end of October 2015 to October 2017, for the research site, were obtained from the South Dakota Climate and Weather Station.

4.2.5 *Statistical analysis*

The statistical analysis was performed as a randomized complete block design with N_2O and NH_3 fluxes as repeated measures using the PROC GLIMMIX procedure of SAS (SAS-Institute, 2012). All measurements were repeated measures based on the date of sampling since multiple measurements came from the same experimental units. Treatments (MB, MO, UO, and NF) and Year (Year 1 and Year 2) were considered fixed factors, whereas, sampling days (Time) and Block were assumed random factors for replication purposes. The mixed model approach also included the effects of Treatment, Year, Time, Block and interactions between them. Differences were considered significant at $P < 0.05$.

The residuals of the N_2O and NH_3 flux data models were not normal. A lognormal distribution improved the Q-Q plots and was used for mixed model analysis. Various covariance structures, such as covariance component (VC), compound symmetry (CS), auto-regression (AR(1)), unstructured (UN), and Toeplitz (TOEP) were then evaluated, and the VC covariance structure selected based on the smallest Akaike information criterion (AIC) value. The obtained least square means (lsmean) from lognormal distribution were back transformed for presentation. For post hoc test, Tukey's Honest Significant Difference (HSD) was used.

4.3 Results and discussions

4.3.1 *Weather condition*

Maximum and minimum air temperatures were 19 and 7ºC in Year 1 and 15 and -1ºC in Year 2, respectively, on the day of manure/fertilizer application. Afterwards, air temperature declined from November to January and then began to increase. Air temperatures in late April were higher than they were in November (Figure 4.2 and 4.3). In Year 1, there was not any rainfall for 12 days after N application; however, rainfall occurred 3 days after N application in Year 2.

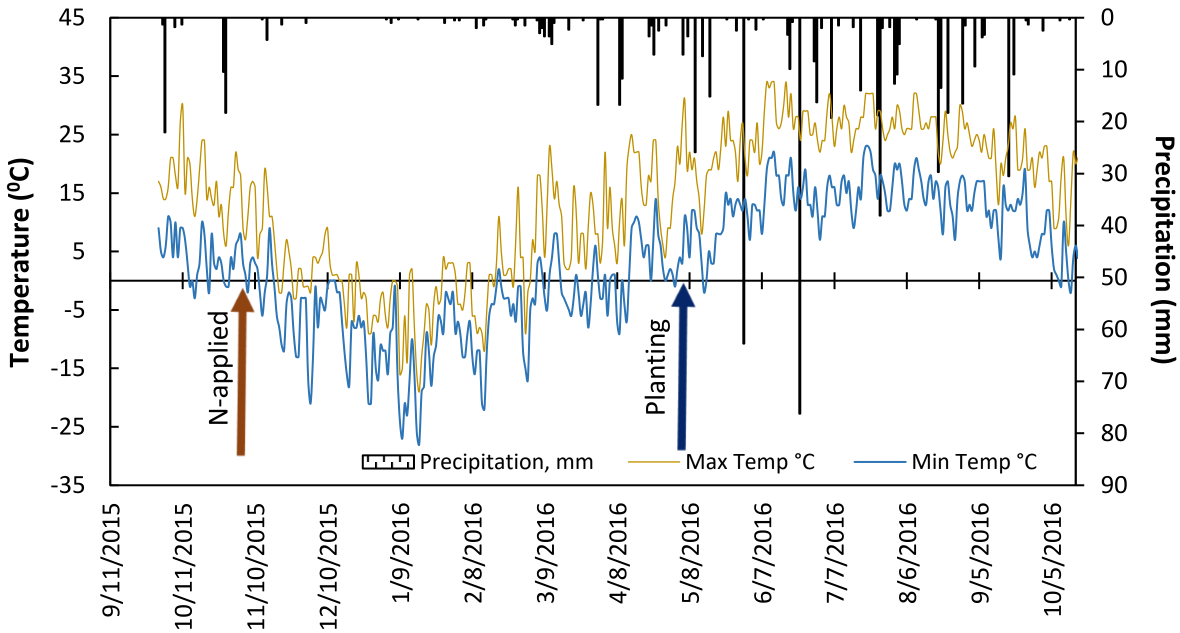

Figure 4.2 Minimum and maximum daily air temperature with daily precipitation from Oct. 2015 to Oct. 2016 (Year 1). The first arrow indicates the N application day, whereas second arrow represents the day of planting.

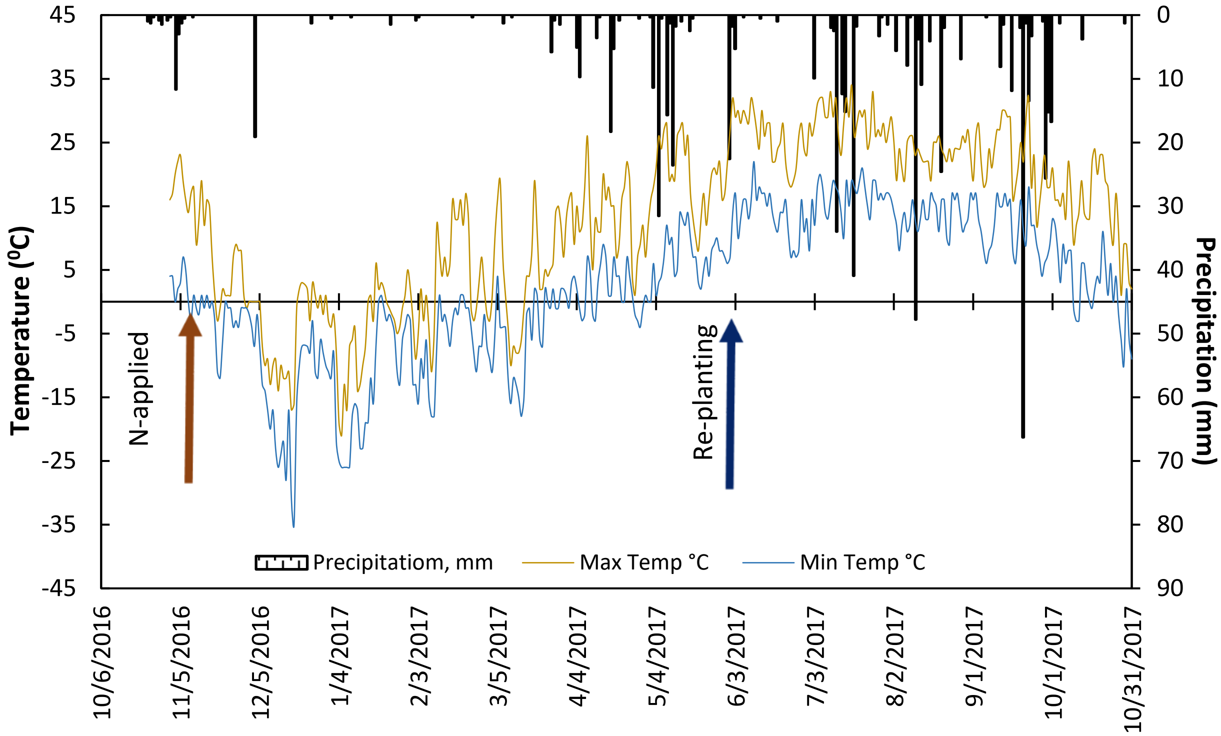

Figure 4.3 Minimum and maximum daily air temperature with daily precipitation from Oct. 2016 to Oct. 2017 (Year 2). The first arrow indicates the N application day, whereas second arrow represents the day of planting.

4.3.2 *Ammonia flux*

The average soil NH_3 fluxes before N applications were 0.69 and 3.3 g ha^{-1} h^{-1} for Year 1 and Year 2, respectively. Table 4.1 shows the average NH_3 fluxes for sampling days after N application for Year 1 and Year 2. We observed NH_3 fluxes increased the first sampling day after N application in the fall for Year 1 and Year 2, however, the fluxes were decreased after the first sampling day from manure plots in both years during fall sampling days (Table 4.1). Also, we noticed that NH_3 fluxes from MB were higher during the first sampling day in both years.

Table 4.1 Average ammonia fluxes (g ha^{-1} h^{-1}) after nitrgoen application for the four Treatments based on sampling day (Time) for Year 1 and Year 2.

Year	Sampling day	Treatment[z]				Mean
		MB	MO	UO	NF	
Year 1	11/6/2015	10.41	1.84	2.09	0.64	3.75
	11/10/2015	8.74	1.76	1.7	1.23	3.36
	11/16/2015	5.23	1.32	1.83	1.16	2.39
	4/26/2016	1.44	1.36	1.57	1.49	1.47
	5/12/2016	1.39	1.36	1.26	1.21	1.31
	6/1/2016	1.62	1.46	1.36	1.59	1.51
	Mean ±SE[y]	3.87±1.28	1.49±0.49	1.63±0.54	1.17±0.39	1.79±0.47
Year 2	11/17/2016	14.59	6.12	4.74	5.17	7.66
	11/22/2016	5.87	3.58	9.84	1.7	5.25
	12/1/2016	2.98	0.65	4.06	0.64	2.08
	4/28/2017	1.49	1.22	1.14	1.45	1.33
	5/16/2017	1.69	1.67	1.60	1.57	1.63
	6/9/2017	1.37	1.35	1.40	1.38	1.38
	7/14/2017	2.13	2.14	1.94	1.98	2.05
	Mean ±SE[y]	3.11±1.03	1.99±0.66	3.03±1.01	1.79±0.59	2.36±0.63

[y]Mean ± SE = Estimated mean ± Standard Error obtained from Year * Treatment Least Squared means table;

[z] MB = solid beef cattle manure with bedding; MO = solid beef cattle manure only, UO = urea only; NF = no-fertilizer.

The analysis showed a significant effect of treatment on NH_3 flux ($P < 0.05$). However, the Year and interaction between Year and Treatment were not significanlty factors for NH_3 flux from the soil surface. The average NH_3 flux from MB was only significantly different than NF, whereas MO and UO were not significantly different from either MB or NF (Figure 4.4). The highest mean (±SE) NH_3 flux was 3.4 ± 0.9 g ha^{-1} h^{-1}, obtained from MB, whereas the lowest mean (±SE) NH_3 was 1.4 ± 0.4 g ha^{-1} h^{-1}

from NF. Adviento-Borbe et al. (2010) reported the NH_3 fluxes were below 1.07 g ha^{-1} h^{-1} (107 µg m^{-2} h^{-1}) from liquid dairy manure and fertilizer N treated plots under corn-corn and corn-alfalfa rotation. Furthermore, they observed the highest soil NH_3 fluxes immediately after manure application, and fluxes were lower thereafter. They stated that decreasing NH_3 flux might be due to decreased total ammoniacal N (TAN= NH_3+ NH_4^+) at the soil surface, infiltration of slurry into soil profile, and a drop in pH due to NH_3 volatilization. The pattern of NH_3 fluxes in our study were similar with them after N application, however the NH_3 fluxes were slightly larger in value. Application timing, methods, N sources, bedding material also affect reported soil NH_3 flux data compared to our study.

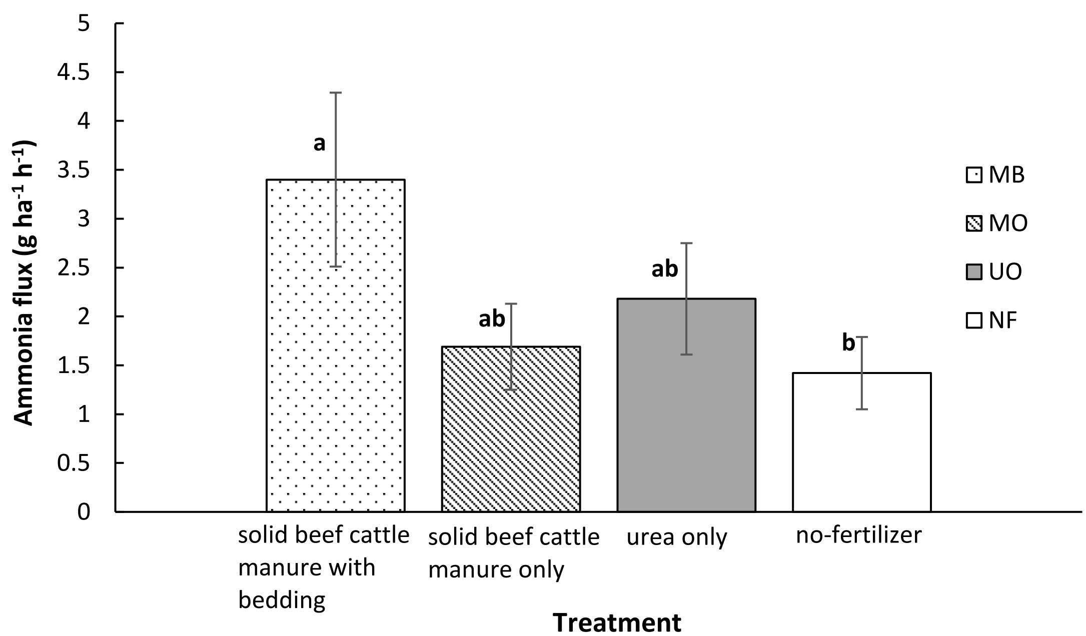

Figure 4.4 Average soil ammonia flux with standard error (vertical lines) from solid beef cattle manure with bedding (MB), solid beef cattle manure only (MO), urea only, and no-fertilizer (NF) treatments for two corn growing seasons (2015/16 and 2016/17). The letters above the bars denote the Least Squared-Means differences at P < 0.05.

The higher NH_3 fluxes from MB treatment could be due to higher ammonium-N (NH_4^+-N) in the manure with bedding compared to the manure only (MO). Huijsmans et al. (2003) observed that soil NH_3 flux increased with an increase TAN in manure. The highest NH_3 flux is often related to the highest manure TAN (Adviento-Borbe et al., 2010; Huijsmans et al., 2003; Miola et al., 2014).

4.3.3 *Nitrous oxide flux*

The N_2O flux samples were collected through the monitoring period after N application and corn planting period, excluding winter and early spring. We wanted to observe the effect of fall-applied solid manure with and without bedding and urea fertilizer on N_2O flux. The Table 4.2 showed the average nitrous oxide fluxes in each sampling day (Time) for Year 1 and Year 2.

Table 4.2 Average nitrous oxide fluxes (μg N_2O-N m^{-2} h^{-1}) after nitrgoen application for the four Treatments based on sampling day (Time) for Year 1 and Year 2.

Year	Sampling day	Treatment[z]				Mean
		MB	MO	UO	NF	
Year 1	11/10/2015	123.40	96.94	105.79	76.81	100.74
	11/16/2015	85.97	26.02	76.61	54.36	60.74
	4/24/2016	580.54	65.53	268.69	103.76	254.63
	5/9/2016	139.27	36.07	42.73	51.48	67.39
	6/1/2016	110.28	5.04	82.21	46.12	60.91
	7/7/2016	101.85	96.58	106.83	90.14	98.85
	8/2/2016	-2.75	7.03	-1.50	0.90	0.92
	Mean ±SE[y]	85.37±39.45	33.81±15.31	79.47±36.72	42.72±19.55	53.71±18.23
Year 2	11/17/2016	40.16	39.64	10.01	1.44	22.81
	11/22/2016	45.84	14.04	18.86	8.89	21.90
	12/1/2016	49.36	111.22	54.18	1.29	54.01
	4/25/2017	57.69	11.49	284.07	8.86	90.53
	5/16/2017	335.32	196.84	416.38	289.39	309.48
	6/9/2017	35.17	35.45	109.77	51.64	58.01
	7/10/2017	69.94	79.46	296.09	62.02	126.87
	8/10/2017	15.65	11.55	54.88	54.26	34.08
	Mean ±SE[y]	30.62±13.04	35.82±15.83	87.57±38.85	15.44±6.96	33.52±10.71

[y]Mean ± SE = Estimated mean ± Standard Error obtained from Year * Treatment Least Squared means table.

[z] MB = solid beef cattle manure with bedding; MO = solid beef cattle manure only, UO = urea only; NF = no-fertilizer.

The analysis showed average N_2O fluxes were significantly affected by treatments ($P < 0.05$). However, only flux from UO was significantly different compared to NF (Figure 4.5). The average (± SE) N_2O flux from UO was 79.6 (±24.9) μg N_2O-N m^{-2} h^{-1}, whereas from NF, it was 24.6 (±7.7) μg N_2O-N m^{-2} h^{-1}. The N_2O fluxes from manure treated plots were not significantly different than UO and NF (Figure 4.5). The average flux (±SE) from manure treatments MB and MO were 49.0 (±15.1) and 33.3 (±10.3) µg N_2O-N m^{-2} h^{-1}, respectively. Year and interaction of Year with Treatment did not show any significant effects on N_2O fluxes. The average (±SE) N_2O fluxes were 53.71 (±18.23) µg N_2O-N m^{-2} h^{-1} for Year 1 and 33.52 (±10.71) µg N_2O-N m^{-2} h^{-1} for Year 2.

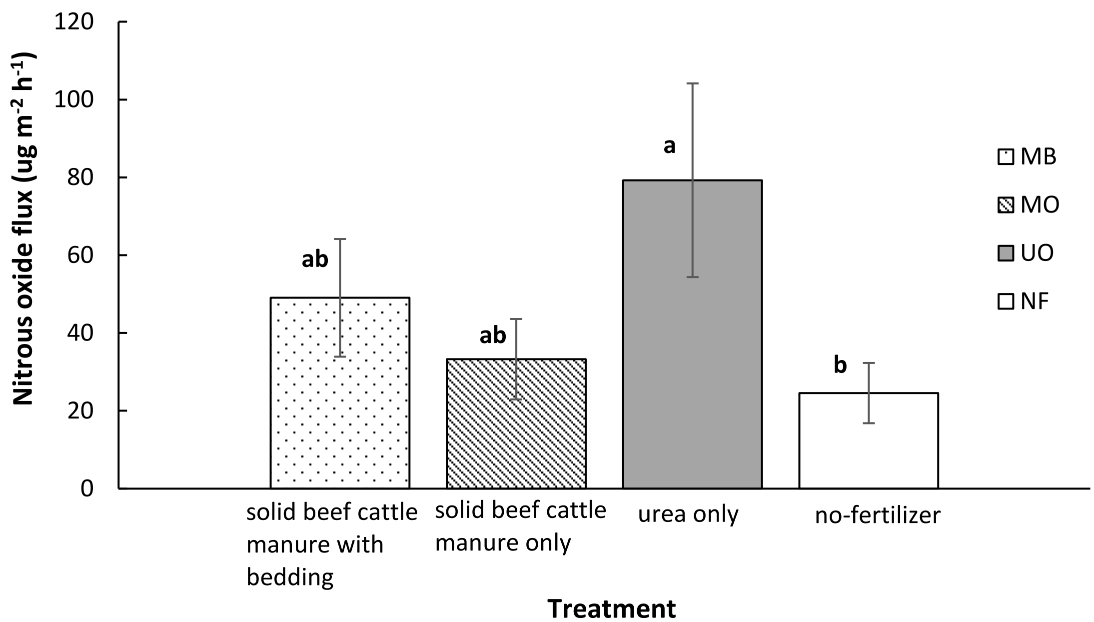

Figure 4.5 Average soil nitrous oxide flux with standard error (vertical lines) from solid beef cattle manure with bedding (MB), solid beef cattle manure only (MO), urea only, and no-fertilizer (NF) treatments for two corn growing seasons (2015/16 and 2016/17). The letters above the bars denote the least square means differences at $P < 0.05$.

Miller et al. (2014) conducted a study to compare long-term land application of stockpiled feedlot beef manure with bedding (barley straw and woodchips) on C/N ratio, denitrification, and carbon dioxide emission in southern Alberta, starting in 1998. They annually applied stockpiled feedlot manure with bedding at the rate of 77 Mg (dry weight) ha^{-1} yr^{-1} for 13 to 14 years to a clay loam soil. The measurement for denitrification fluxes were taken in 2011 and 2012 (every 2 weeks between May and August). They found mean N_2O fluxes for manure with straw bedding were between 3.7 and 4491.7 μg N_2O-N m^{-2} h^{-1} (0.9 and 1078 g N_2O-N $ha^{-1}d^{-1}$), from 3.3 to 1358.3 μg N_2O-N m^{-2} h^{-1} (0.8 to 326 g N_2O-N ha^{-1} d^{-1}) for manure with woodchips bedding, and 2.5 and 1041.7 μg N_2O-N m^{-2} h^{-1} (0.6 to 250 g N_2O-N ha^{-1} d^{-1}) for control. However, they observed that total N, daily denitrification flux, and daily carbon dioxide flux were not affected by bedding materials. Akiyama and Tsuruta (2003) measured N_2O flux for poultry manure (PM), swine manure (SM), and urea applied to soil using an automated flux monitoring system. They found the total fluxes were 21, 7, and 5 μg N_2O-N m^{-2} h^{-1} (184, 61.3, and 44.8 mg N_2O-N m^{-2} y^{-1}) from PM, SM, and urea, respectively.

Engel et al. (2010) studied the effect of urea placements (broadcast, band, and nest) on N_2O emission from a silt loam soil. The rate of urea application was 200 kg N ha^{-1}. They found maximum N_2O flux for broadcast surface, broadcast incorporated, band, nest, and control were 61.7, 55, 103.3, 117.1, and 12.9 μg N_2O-N m^{-2} h^{-1} (14.8, 13.2, 24.1, 28.1, and 3.1 g N_2O-N ha^{-1} d^{-1}), respectively. The maximum mean N_2O flux (79.6 (±24.9) μg N_2O-N m^{-2} $h^{-1)}$ in our study was very low compared to the maximum fluxes reported by Miller et al. (2014), it might be because of lower rate of solid beef manure application (about half application rate), different bedding material used (corn Stover vs barley straw and woodchips), different gas measurement technique (Static Chamber derived by Parkin and Venterea, 2010 vs gas measured by using Chang et al. (1998) method), and weather condition. However, our study showed higher N_2O fluxes than the fluxes reported by Akiyama and Tsuruta (2003), and this difference is likely related to soil properties (Silty clay loam vs Andisol (Volcanic ash soil)), types and rate of N sources, sampling method, and different climatic condition. In contrast, the N_2O flux obtained for broadcast incorporated N placement by Engel et al. (2010) study, was similar to N_2O fluxes from urea treated plots from our study.

4.4 Conclusions

A two-year field study was conducted to investigate NH_3 and N_2O fluxes from silty clay loam field after application of manure with and without bedding and urea in fall. Our study showed that soil N_2O, NH_3 fluxes, and soil water nitrate were not significantly different between solid beef cattle manure (with and without bedding) and urea. The N_2O flux from UO was significantly higher than flux from NF, and NH_3 flux from MB was significantly greater than flux from NF. The N_2O fluxes from manure treated plots were not significantly different with UO and NF. Similarly, NH_3 fluxes from MO and UO were not significantly different than either MB or NF. We observed the NH_3 flux for MB was higher after application of N during the first week of sampling and decreased thereafter. The variation in soil fluxes compared to previous studies could be due to sources of N, manure characteristics (total N, total C, bedding, ammoniacal N), total soil NO_3^--N, available soil ammonium-N, method used for samples collection and flux calculation, soil types, and weather condition. Understanding soil fluxes and affecting factors will help us to minimize the possible N losses as gases forms from soil volume and will help to reduce environmental impact by those soil fluxes.

CHAPTER 5 DISCUSSION AND CONCLUSIONS

5.1 Summary

The study was conducted for comparison of bedded versus non-bedded solid beef cattle manure, as well as urea and no-fertilizer effects on soil NO_3^--N, crop characteristic, crop yield, and soil N losses on the silty clay loam soil type in the Northern Great Plains region. The total soil NO_3^--N, NH_3 flux, and N_2O flux were significantly affected by treatments, whereas soil water NO_3^--N concentration was not significantly different among the fall-applied manures and fertilizer treatments and control. Total soil NO_3^--N was significantly higher for UO treatment compared to others three treatments, whereas the total soil NO_3^--N concentrations were not significantly different for MB, MO, and NF. The average NH_3 flux was significantly greater for MB compared to NF, however, the NH_3 fluxes from MO and UO were not significantly different than MB and NF. Soil N_2O flux for UO was significantly higher than NF, while this fluxes from manure treated plots (MB and MO) were not significantly different from either UO or NF. The study did not show any notable change in soil water NO_3^--N concentration among the treatments from observation between corn emergence stage and V6 stage, although the lowest soil water NO_3^--N concentration was found in MB treated plots. However, the soil water NO_3^--N concentration significantly changed between Year 1 and Year 2. Furthermore, corn leaf-N and grain-N tended to be affected by treatments, however, corn yield was not affected by either treatment in Year 1.

Our study was based on plot-scale sampling, coupled with manual N (manure and urea) application, which was not entirely indicative of full-scale conditions. However, the benefits of tighter control of treatment conditions of our study provide a more accurate dataset for comparative analyses and model application. Our study helped to document the effects of solid beef cattle manure with and without bedding on various path of soil N losses and corn N characteristics. The secondary use of our study data is for modeling purposes, such as in the Integrated Farm System Model (derived by C.A. Rotz). Modeling helps to further understand factors and processes affecting nutrient transformations and release during corn production with beef cattle manure fertilizer. The ultimate selection of management practices by producers is based on many factors including environmental losses, climate and soil factors, and economic conditions.

5.2 Lesson learned from this research and future work

The results from this research enhances the understanding of N loss mechanism and transformation processes in silty clay loam soil. Furthermore, the research helps us understand how the nitrogen source, or treatment characteristics, influence N losses. To understand the soil N losses, crop characteristics (leaf-N and grain-N), and yield, it is important to understand the total soil N and the transformation processes and factors affecting them. The study revealed that urea associated plots obtained the highest total soil NO_3^--N concentration, leaf-N, grain-N, and yield. However, only total soil NO_3^--N for urea plots were significantly different than remaining treatments ($P < 0.05$). Soil water nitrate concentrations were not significantly different between any treatments. Soil water nitrate for Year 1 was significantly higher than Year 2. Our study depicted that soil water nitrate concentration depends on rainfall and available soil nitrate.

The study reinforced that NH_3 fluxes are higher after N application and similar for all N treatments during the corn growing period. However, the amount of NH_3 flux was dependent on the rate of N, weather and soil conditions.

The N_2O fluxes in fall were lower than in spring. There was no significant difference between manure and urea treatments. However, urea treated plots showed significantly higher N_2O flux than no-fertilizer. The N_2O flux appeared to vary based on plant-available N. The flux correction method accounted for the potential effects of soil temperature and soil moisture.

From the two-year study, we captured the manure and fertilizer effects on total soil NO_3^--N, N losses, leaf-N, grain-N, and yield under corn production. These information helps us to select the management practice that release less soil N, however each management practices have their own additional pros and cons.

In the future, the study can be improved for greenhouse gas measurement by using automated gas measurement techniques. Manual static gas chamber methods can alter soil and microclimatic conditions and provide low temporal frequency data (Yao et al., 2009). Bias can be reduced by frequently changing the position of chambers and/or opening the chambers automatically during rainfall events (Yao et al., 2009). Automated measurement techniques can take automatic continuous or near-continuous measurements which may improve flux estimation and capture diurnal variations (Flessa et al., 2002; Yao et al., 2009). The static chamber method used in our research provided a means of comparing flux based on nitrogen application treatment. In the future, use of automated chamber techniques will further the quantification of diurnal and annual cumulative soil fluxes for manure management practices in the Northern Great Plains.

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