Evaluation of Seasonal Dynamics of Soil Macro-Nutrients and Corn Nutrient Uptake in Fields Amended with Three Types of Municipal Biosolids

by

Zheya Lin

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ABSTRACT

Land application of biosolids as a source of crop fertility and soil organic matter can be viewed as a sustainable approach to maintain soil productivity. Plant growth can be partially supported through the use of these amendments, thereby minimizing reliance on chemical fertilizers. Field-based experiments are being conducted to examine the effect of three municipal biosolids (composted, liquid mesophilic anaerobically digested, and alkaline stabilized) on soil nutrient dynamics, specifically N, P, and K, applied over three years in Nova Scotia. A total of fifteen treatments are being evaluated in this study based on management associated with surface vs. incorporation of biosolids and the application of biosolids with urea supplementation. Additional agronomic parameters, including crop yield and plant nutrient uptake, are being examined relative to the amendment rates to develop nutrient use efficiency indices. The results suggest that applying municipal biosolids can increase nutrient availability. Alkaline treated biosolids can increase soil pH in acidic agricultural soil. The incorporation of biosolids was greater than the surface spreading on soil mineral nitrogen, crop yield, and nitrogen uptake, but no management practices and rate effects were observed on Mehlich 3 extractable P and K and plant P and K uptake in this study. Crop responses highly depended on N availability and weather conditions and were greatest for alkaline treated biosolids > liquid mesophilic anaerobically digested biosolids > biosolids compost.

LIST OF ABBREVIATIONS USED

AE	Agronomic efficiency
ANOVA	Analysis of variance
ANR	Apparent nutrient recovery efficiency
ATB	Alkaline treated biosolids
AUC	Area under curve
CaO	Lime
CH_3COOH	Acetic acid
CKD	Cement kiln dust
COMP	biosolids compost
ICP-OES	Inductively coupled plasma optical emission spectrometry
K	Potassium
LMAD	Liquid mesophilic anaerobically digested biosolids
M3K	Mehlich 3 extractable potassium
M3P	Mehlich 3 extractable phosphorus
MP	Management practices
N	Nitrogen
NS	Nova Scotia
NUE	Nutrient use efficiency
P	Phosphorus
PTK	Plant total potassium
PTN	Plant total nitrogen
PTP	Plant total phosphorus
SMN	Soil mineral nitrogen
SNR	Soil nitrogen ratio
SOM	Soil organic matter
TN	Total nitrogen

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1. Introduction

1.1 Overview

Biosolids are a nutrient-rich by-product of wastewater treatment plants, containing a significant amount of nitrogen (N) and phosphorus (P). Land application of biosolids for crop fertility is an alternative approach to dispose of the waste, as well as to improve and maintain productive soils. Soil conditioners such as biosolids can provide plant nutrients, thereby minimizing the reliance on chemical fertilizers (National Research Council, 2002; Lapen et al., 2008). Increasing global populations will continue to generate sewage solid wastes that require treatment and management. Approximately 4 billion tons of sewage are produced worldwide each year, Canada currently generates more than 205 billion liter sewage and around 660,000 dry tons of biosolids each year from its wastewater treatment plants, but only 33% of the total biosolids produced in Canada are applied to agricultural lands (CCME, 2012; Thompson, 2016).

The Canadian government has developed policies to deal with the growing amount of sewage solids by regulating the treatment processes for biosolids and developing guidelines for the appropriate uses of biosolids. Public concern for the land application of biosolids continues to rise nationally (Wang, 1997; Wang et al., 2008; CCME, 2012). Although well-established practices of applying biosolids on land exist, some scientific literature is focused on the waste management perspective rather than nutrient recovery, while other researchers have focused on agronomic practices. Many scientific studies on the land application of biosolids have been published, but management strategies to optimize nutrient recovery of biosolids in cropping systems are typically site-specific, based on local climatic conditions, soil properties, and biosolids treatments. Furthermore, the effect on the nutrient use efficiency (NUE) of crops from biosolid treatment technologies, including anaerobic digestion, alkaline stabilization, and composting, is poorly understood. Therefore, consideration of the composition of biosolids is critical in developing sustainable nutrient management practices to support crop production and maintain soil fertility.

1.2 Literature review

1.2.1 Biosolids background

Municipal wastewater includes domestic sewage, industrial wastewaters, and primary rain runoff into drainage canals. Domestic sewage mainly contains fecal waste generated by humans, as well as other components such as pharmaceuticals, food wastes, soaps, and oil grease. To meet the requirements for land application or disposal, these materials still need further treatment to reduce the pathogen content and remove other contaminants to achieve what is currently termed biosolids (Fig. 1.1)(CCME, 2012). Conventionally accepted treatment processes to generate biosolids include anaerobic digestion, composting, alkaline stabilization, and heating (Nova Scotia Environment, 2010). Biosolids are a nutrient-rich product consisting of organic compounds, a variety of macronutrients, and micronutrients that can be used in agriculture (Singh & Agrawal, 2008). The available macronutrients in biosolids can be used to improve the soil properties and stimulate plant growth and, in many instances, increase crop yield (NRC, 2002).

In Canada, around 50% of the total biosolids produced are land applied, and energy generation accounts for 47%, while 4% are sent to landfill (Apedaile, 2001; EDI Environmental Dynamics Inc., 2017). Biosolids land application is covered under different regulations and guidelines based on each province in Canada. In Quebec, biosolids, which are sold as a fertilizer, are regulated under the Agriculture and Agri-food Canada Fertilizer Criteria and the Bureau de

Normalization du Quebec Fertilizer Regulation. In Nova Scotia, biosolids can be divided into two categories, Class A and Class B, according to different quality criteria (trace elements, heavy metals, pathogen reduction, and odor reduction) (CCME, 2010). Class A biosolids are treated and stabilized to a high standard for pathogen content and heavy metals (Nova Scotia Department of Environment, 2010). In contrast, Class B biosolids receive a lower level of treatment and have higher allowable heavy metal concentrations. Therefore, land application of Class B requires special approvals from the Nova Scotia Department of Environment, and this type of biosolid cannot be applied to agricultural land. Class A biosolids are unrestricted and can be applied to agricultural land with no further approvals or monitoring requirements (Nova Scotia Department of Environment, 2010).

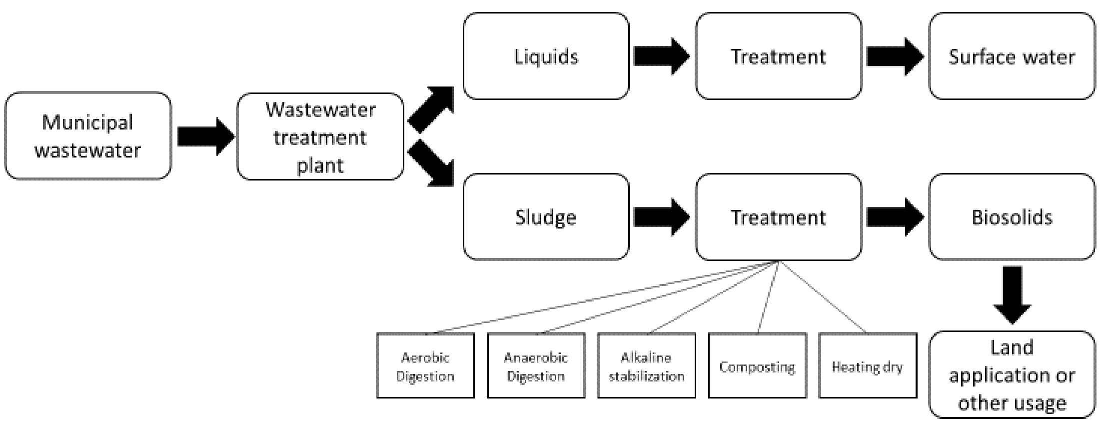


Fig. 1.1 Municipal wastewater treatment process and alternative reuse methods

1.2.1.1 Biosolids compost

Biosolids compost is a humus-like substance without detectable levels of pathogens, and the organic matter is decomposed under aerobic thermophilic conditions in porous and size-controlled piles (U.S. EPA, 2002). This process involves a mixture of dewatered biosolids and various bulking agents, such as sawdust and woody residuals, which causes the aerobic decomposition to occur over time. During the composting process, microorganisms consume organic matter and generate heat. As the temperatures increase, some microbial activity increases, but many organisms and pathogens are killed. Bulking agents improve oxygen flow and moisture control for microbes, maintaining a steady-level of airflow. After the primary composting stage, the compost cures for an additional period, which further stabilizes the mixture (NEIWPCC, 2001; Shammas & Wang, 2007). Three different methods are commonly used to compost sewage solids to generate a biosolids compost, including windrow composting, aerated static piles, and in-vessel systems. These follow the same composting principle but differ in management and equipment requirements. (U.S. EPA, 1999; NEIWPCC, 2001).

The final biosolid product is Class A and Class B based on the treatment conditions and can be used as a soil conditioner and fertilizer for gardens and feed crops. Composts usually have a pH ranging from 6.5 to 8. One organic amendment was applied at 0, 75, 150, and 300 m^3 ha^{-1} to loamy and clay soils consisting of 62% composting municipal waste, 21% sewage sludge, and 17% sawdust, and found organic amendments have a significant amount of organic matter (32%), improve aggregate stability and soil cation exchange capacity as well as nutrient release to the soil (Aggelides & Londra, 2000). Zubillaga and Lavado (2002) reported a 40% increase in lettuce production, adding 50% to 75% biosolids compost. Cu and Ni concentrations in lettuce were positively correlated with the increase in the rates (0, 25%, 50%, 75%, and 100%) of biosolids compost applied in Buenos Aires, Argentina. Results from Mantovi et al. (2005) suggested an increase of organic matter content in topsoil with biosolids compost over 12 years at 5 to 10 Mg DM ha^{-1} yr^{-1} in winter wheat–sugar beet–maize rotation. Moreover, they found that biosolids compost increased the N and P content of the wheat grain, N and Cu content of sugar beet, and Cu content of corn. Repeated biosolids compost applications in a Colorado semi-arid grassland increased soil NO_3-N and NH_4-N and decreased AB-DTPA extractable Ba (Ippolito et al., 2010). Biosolids compost application also increased yields in barley and Chinese cabbage production systems (Wei & Liu, 2005). However, biosolids compost has some drawbacks, such as odor problems during production and unstable compost quality if not processed to maturity (Shammas & Wang, 2007).

1.2.1.2 Alkaline stabilization of biosolids

Alkaline stabilization, also referred to as alkaline treated biosolids (ATB), is a biosolids treatment method that is one of the approved methods before land application in Canada (Nova Scotia Department of Environment, 2010). The N -Viro process is commonly used in Eastern Canada and has a special drying process on the top of the admixture (N-Viro System Canada LP, 2009). The process can be described as the drying of dewatered sewage sludge with the addition of an alkaline admixture to bring the pH up to 12 and raise the temperature (52 to 62°C) to kill pathogens. Most alkaline admixtures are industrial by-products, which are low cost, including cement kiln dust (CKD), limestone, lime kiln dust, quicklime (CaO), hydrated lime, fly ash, wood ash, and other coal-burning ashes (U.S. EPA, 1999). They can be used in these treatment processes to destroy pathogens under a high pH environment (Wong & Selvam, 2009). The ATB generated in Nova Scotia is classified as a Class A biosolids and provides a reasonable pH adjustment in agricultural soils, especially in acidic soils (Price et al., 2015). Moreover, the organic matter associated with ATB also improves other soil properties, such as water holding capacity (U.S. EPA, 2000).

The ATB product has been applied to agricultural land for pH adjustment and as a fertility alternative (Bigham et al., 1999). Christie et al. (2001) reported an increase in soil pH using ATB, resulting in lower shoot Mn concentrations in a spring barley field and higher crop yields. They also found that the ATB had the same amount of available P and K as conventional fertilizer. Wong et al. (2004) applied 4% ATB (w/w) in acidic, loamy soil to grow mustard for site remediation, resulting in higher biomass and extracted cadmium from plants than control. Da Silva et al. (2011) demonstrated that the effects of ATB added three levels of phosphorus to test the acidity and exchangeable cations in Oxisols. Under these experimental conditions, ATB in acidic Oxisol had the maximum increase of soil pH, exchangeable calcium and magnesium, and a reduction of exchangeable aluminum and potential acidity. Even though the short-term effects of ATB to increase soil pH may vary, a small amount of heavy metals still leach into the soil profile (Luo & Christie, 2001; McBride, 2003). A four-year field study examined the annual application of ATB from N-Viro at a high rate ($\geq$ 14000 kg ha^{-1}) increased the seasonal soil mineral nitrogen by 15 to 42% and soil pH by 1.5 pH units, and it also had an 8.4% increase in soil total P and enhanced the crop productivity (Price et al., 2015; Shu et al., 2016).

1.2.1.3 Mesophilic anaerobic digestion of biosolids

Anaerobic digestion stabilizes sewage sludges under the influence of anaerobic bacteria, converting the organic solids into CO_2, CH_4, and NH_3 in a closed tank to reduce the organic content, odor, and pathogen levels in biosolids (Appels et al., 2008). Anaerobic digesters are commonly operated in a mesophilic temperature range (35-38°C) or a higher thermophilic temperature range (50-65°C). Class B biosolids are generated with mesophilic digestion, and mesophilic anaerobic digestion is a standard system that has lower energy demand and a lower biogas production rate but longer retention time compared to the thermophilic system (Gebreeyessus & Jenicek, 2016).

The treatment process is divided into two phases, and these are the acid phase and the methane fermentation phase. In the first phase, the acid-forming bacteria will convert the complex organic compounds into simpler organic compounds, followed by organic acids, which are mainly acetic acid (CH_3COOH). The pure soluble organics produced during the hydrolysis stage are further decomposed into volatile fatty acids, alcohols, ketones, aldehydes, carbon dioxide, and hydrogen under the action of hydrogen-producing and acid-producing bacteria (Mata-Alvarez et al., 2000). After the first phase, methanogens convert CH_3COOH, H_2, and CO_2 to methane according to the different chemical reactions (Labatut et al., 2014). The stabilization of organic waste mainly occurs in the second stage. Anaerobic digestion can lead to a high degree of waste stabilization and reduce pathogens, volume, and organic matter, as well as improve sludge dewaterability (Taricska et al., 2007).

The end product of mesophilic anaerobic digestion of sewage sludge is Class B biosolids with a high concentration of nitrogen (low C: N ratio), which can be applied as a nutrient source. Many crops, including corn, ryegrass, and legumes, have been grown successfully, with higher yields using anaerobically digested biosolids than control (Bougnom et al., 2012; Gagnon et al., 2012; Fouda et al., 2013). A one-year experiment of a degraded semiarid ecosystem in Spain was conducted to determine the effect of anaerobically digested biosolids (40, 80, and 120 Mg ha^{-1} dry wt.) on soil chemical properties, total plant cover, and total aboveground biomass. There were little impacts of the biosolid amendment on soil chemistry, but the concentrations of available P and NO_3-N significantly increased after application, and a small amount of heavy metal content also increased (Walter et al., 2000). The results of Haney et al. (2015) indicated that after eight-year continuous annual surface applications of anaerobically digested biosolids on forage production in central Texas, the concentration of inorganic N and P increased linearly with application rate (0, 20, 40, or 60 Mg dry biosolids ha^{-1}), however, the distribution of soil K has no impact on biosolid applications. Pathogen and odor concerns may be more severe after anaerobically digested processing during land application. Anaerobically digested biosolids with high levels of pathogens, often as Class B biosolids, may reduce the area of land available for agronomic applications and are applied in areas as a restricted use for remediation, i.e., mining site (Sahlström, 2003; U.S. EPA, 2006; Sheets et al., 2015).

1.2.2 Soil fertility

1.2.2.1 The soil in Nova Scotia

Nova Scotia (NS) is one of the four most eastern provinces in Canada on the Atlantic coast. Due to the influence of the Atlantic Ocean, soils in Nova Scotia have formed under a moist climate. Most soils in the region are classified as podzolic according to the Canadian Soil Classification System and are characterized primarily with sandy loam textures (Canada Soil Landscape, 2016). Podzols can occur on many parent materials, but mainly from quartz-rich sandstones, sandstones, and sedimentary debris from magmatic rocks, which have high precipitation. Podzols are often acidic soils, which are inadequate for agricultural production, resulting in low nutrient availability (Mokma et al., 2004; Gupta et al., 2008). In addition, high precipitation results in severe leaching of elements, such as calcium, magnesium, and potassium from the surface soil, making it strongly acidic. Aluminum and manganese are more soluble in acidic soil and can be present in toxic amounts in solutions surrounding soil particles and roots. Fertilizer efficiency is reduced, and the activity of soil bacteria is significantly limited. Therefore, soil acidity is a primary limiting factor for crop production in many parts of Atlantic Canada, including Nova Scotia (Forestry and Agrifood Agency, 2010).

1.2.2.2 Nutrient forms in soil

Soil nutrients are chemicals essential for plant growth provided by the soil and its constituents, including the macronutrients (N, P, K, S, Ca, and Mg) and micronutrients (Zn, Cu, Fe, Cl, B, Mn, and Mo). The three elements (C, N, and P) are believed to play a critical role in soil fertility and plant growth. The content of C determines the soil organic matter (SOM) content, with higher organic C inputs stimulating microbial activity and supporting the release of other nutrients (Havlin et al., 2016).

The nitrogen cycle is one of the most critical nutrient cycles in the soil, and total N concentration ranges from <0.02% in the subsoil to >2.5% in organic soil. In surface soil, more than 90% of the soil's total N is present in organic compounds (Bremner & Mulvaney, 1982). Only 2 to 5% of total N in soil is in the form of NH_4^+ and NO_3^-, which is available for plant uptake. Nitrogen mineralization, through the hydrolysis and biodegradation of organic matter, can provide sufficient NH_4^+ and NO_3^- to meet plant requirements from the soil organic N pool (Pierzynski et al., 2005). The amount of available N in plants released from organic N depends on many factors that affect N mineralization, immobilization, and loss of NH_4^+ and NO_3^- from the soil. Inorganic N consists of different forms such as NH_4^+, NH_3, $NO_2^-$and NO_3^-. NH_4^+ can be divided into exchangeable NH_4^+, which is generally less than 30% of soil inorganic N (Stevenson, 1982).

While only 0.005 to 0.15% of the total P in the surface soil and declines with increasing weathering intensity (Havlin et al., 2016). Phosphorus has two major forms in soil, including organic P and inorganic P. Inorganic P consists of orthophosphate, pyrophosphate, and polyphosphate. Orthophosphate is found as $H_2PO_4^-$ and HPO_4^{2-} in the soil solution; they are both available P forms for plant uptake (Gary et al., 2004; Pierzynski & McDowell, 2005). In the neutral pH range, both forms of P are present in equal proportions. However, $H_2PO_4^-$ dominates under acidic conditions, and the concentration of HPO_4^{2-} increases when pH is higher than 7.2

but below 12. About 50% of the total P in soil is in the organic form. If adding OM to the soil, the percentage of organic P will increase, but its availability decreases with depth (Condron et al., 2005). The mineralization of organic P leads to inorganic P in the soil. Inorganic P is a usable form of soil P that can only be used for plants if it does not adsorb onto the mineral surface of the soil (Pierzynski & McDowell, 2005; Havlin et al., 2016).

1.2.3 Nutrient availability from biosolids

The N content in biosolids can vary greatly, ranging from 1 to 6%, and most biosolids include organic N (around 80% of total nitrogen), NH_4^+-N, and NO_3^--N (Havlin et al., 2016). The concentration of inorganic N in biosolids is between 0 to 50% of the total N concentration, and most inorganic N exists in the form of NH_4^+-N, while NO_3^--N exists at lower concentrations (Rigby et al., 2016). NH_4^+-N can be directly available to plants after application, but organic N must be converted into inorganic N via nitrogen mineralization before being available to the crops. The total content of organic N and NH_4^+-N in a biosolid depends on the stabilization process. For example, liquid anaerobically digested biosolids usually have more NH_4^+-N than organic N. While in the heat-dried biosolids, over 90% of N is organic, and a small amount is NH_4^+-N (Haynes et al., 2009; Sullivan, 2015). MAD biosolids from the USA and Australia had larger inorganic N proportions relative to total N, ranging from 28.5 to 45.4% (Al-Dhumri et al., 2013; Evanylo & Sullivan, 2003). While the inorganic N content of MAD in the UK only ranged from 12.5 to 17.3% (Morris et al., 2003; Rigby et al., 2009). The inorganic N content of ATB ranges from 2.6 to 65.1%, while the smallest amount of inorganic N is from 10 to 24.5% for biosolids compost (Rigby et al., 2016).

The phosphorus content in biosolids varies from 2 to 4% on a dry weight basis, with 90% of the total P in inorganic forms (Smith et al., 2006; Havlin et al., 2016). There is plenty of Fe, Al, Ca, and Mn in biosolids to bind P, which then becomes the dominant form of inorganic P in soil (Frossard et al., 1996). Activated and aerobically digested biosolids had a higher concentration of organic P than anaerobically digested biosolids. Analysis of soils using ^{31}P NMR and an enzyme assay procedure showed different proportions of organic fractions (orthophosphate diesters and monoesters) in different biosolids (Hinedi et al., 1988). Smith et al. (2006) determined the P dynamics of three types of sewage sludge over 151 days of incubation. They found that different sources and treatment processes for sewage sludge resulted in high variability in both P concentrations and forms. During land application, people have to apply a significant amount of P in biosolids to meet the requirement of crop N, and the supplemental P can have long-term benefits for soil fertility in deficient soils (Sullivan, 2015). However, high P content in soil may increase the potential rate of P loss (leaching and runoff) to surface waters, leading to eutrophication and the reduction of water quality (Sullivan, 2015; Shu et al., 2016).

1.2.4 Nutrient availability in biosolid-amended soil

Nutrient availability in crop production and the timing of the nutrient release in biosolid-amended soils are affected by biosolid properties, climate, vegetation, soil microbial community, and the characteristics of soil (Sullivan, 2015). Cogger et al. (2001) applied municipal wastewater biosolids for seven years at annual rates of 290, 580, and 870 kg ha^{-1} total N with tall fescue, which maintained high yields and increased soil nitrate at the highest application rate. P content in biosolid-amended soil was also high (>400 mg P kg^{-1} soil) in all treatments. Similar results were also obtained in a subsequent two-year biosolids application study, which also found that N uptake had a positive linear relationship with the application rate (Cogger et al., 2004). Shober et al. (1996) tested the nutrient contents of soil in 18 production farms in Pennsylvania for long-term effects on which the application rate of biosolids ranged from 5000 to 159000 kg

ha^{-1} on a dry weight basis. They reported higher concentrations of NO_3 -N (13.21-26.21mg kg^{-1}), P (156.1-257.7 mg kg^{-1}), Ca, Cu, Mn, and Zn, but less soil K in biosolid-amended soil. A study using five types of biosolids (fresh sludge, composted with hardwood sawdust, composted with woodchips, heat-dried, and lime-treated) to supply N and P to ryegrass found that biosolids compost had the highest N concentration, and around 36% was recovered in plant tissue. The ryegrass grown with fresh and lime-treated biosolids recovered 18% of N, but only 6% of N was recovered from heat-dried biosolids. However, only 3 to 7% of P was recovered in plants with these five biosolids, indicating the P fixation in the soil (R. S. Corrêa, 2004).

1.2.5 Surface spreading vs. Incorporation

It is necessary to consider whether biosolids should be applied to the soil surface or incorporated in the topsoil. There are many implications related to surface spreading vs. incorporation of biosolids, such as fertilizer efficiency, ammonia volatilization, leaching, and risks from the fun-off of pathogens (Gove et al., 2002).

Surface spreading can increase the amount of water that infiltrates through the soil, and it also increases the retention of organic matter and nutrient cycling in soil (Beare et al., 1994). Applying on the surface can reduce nutrient leaching compared to incorporation, but it increases the risk of surface run-off, the loss of N related to ammonia volatilization, and other issues such as odor (Smith, 1995; Girovich, 1996; Nicholson et al., 2004).

Incorporation of biosolids, using approaches such as deep tillage or disking, is one of the oldest management practices to help retain nutrients to enhance plant growth (Martens, 2001). Incorporation decreases nutrient loss, especially for N, which can be lost as NH_3, and also may minimize subsurface water pollution. One report showed that incorporation could save an additional 25% of the total N applied to the soil compared to surface spreading (Forestry and

Agrifoods Agency, 2010).

1.3 Objectives and hypotheses

The overall objectives of this research project are to:

1. Investigate seasonal soil nitrogen dynamics and plant N uptake in relation to the rate and application methods of three types of biosolids following addition to an agricultural soil over three years.
2. Examine seasonal soil phosphorus and potassium dynamics and plant P and K uptake in relation to the rate and application methods of three types of biosolids following addition to an agricultural soil over three years.
3. Determine the nutrient use efficiency of corn as influenced by three types of biosolids applied to agricultural soil for three consecutive years.

This study hypothesized that the three years of continuous application of municipal biosolids would (i) increase the available forms of soil N, P, and K for plant uptake and lead to P and K accumulation in the soil. (ii) Alkaline treated biosolids would have the best performance in all parameters among three municipal biosolids, followed by anaerobically digested biosolids and biosolids compost. (iii) Half rate treatments would have higher NPK content in soil and crop yields compared to full rate treatments. (iv) Incorporated biosolids would result in a higher yield and crop nutrient uptake than surface applied biosolids.

2. Seasonal Soil Dynamics and Plant Uptake of Nitrogen in Corn Fields Amended with Biosolids

2.1 Introduction

Biosolids are solid organic by-products from wastewater treatment plants that undergo treatment for pathogen reduction and stabilization. The production of biosolids in Canada and the United States is around 0.9 Mg and 7.1 million Mg (w/w) each year, and approximately 60% are recycled to agricultural land (Zerzghi et al., 2010). Land application of biosolids not only improves soil quality but recovers nutrients back to the soil. This is an alternative to other disposal methods, such as landfilling or incineration (Kaleeem Abbasi et al., 2015; Rigby et al., 2016).

The primary treatment processes for sewage sludge include anaerobic digestion, composting, and alkaline treatment. The process of alkaline treatment includes drying a mixture of sewage sludge and an alkaline admixture (cement kiln dust, quicklime, alkaline flying ash, and wool ash) to reach a high pH (>12) and temperature (52~62°C) in order to kill pathogens (Logan & Harrison, 1995). Anaerobic digestion typically occurs in a two-stage closed tank system, where bacteria break down the complex solids to organic acids and then convert them to methane (Salsali et al., 2005). Compared to mesophilic anaerobic digestion, the process of composting sewage solids into a biosolid is more straightforward, involving a mixture of dewatered sewage sludge and various carbon and bulking agents, such as sawdust and woody residues under aerobic conditions (Calbrix et al., 2007).

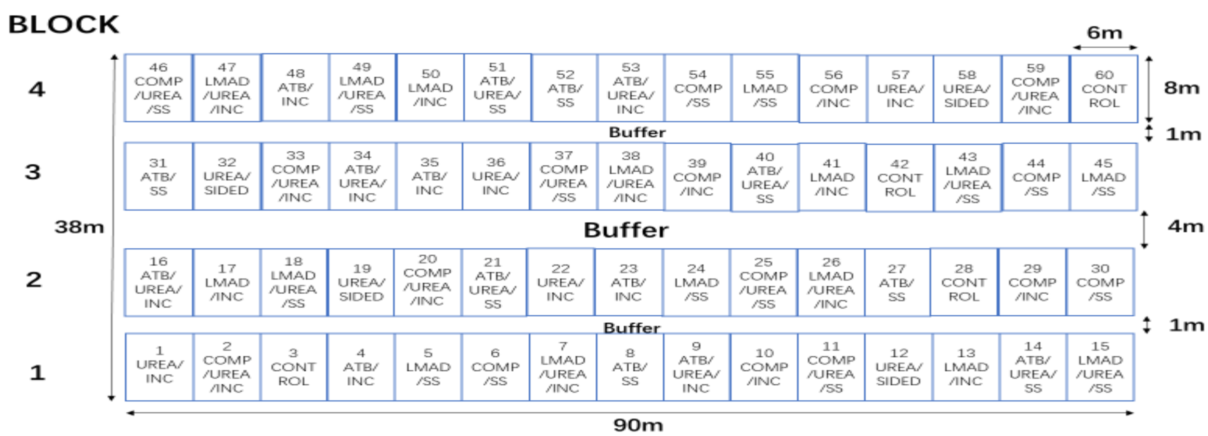
The concentration of total and mineral nitrogen (N) in biosolids and the release of inorganic N into the soil can be significantly influenced by the process used to treat the sewage sludge (Cogger et al., 2004; Pritchard et al., 2010). Liquid mesophilic anaerobically digested (LMAD) biosolids typically have high total N content (TN), in the range of 4.3 to 15% on a dry basis, with inorganic N content accounting for 14.7 to 44.2% of the total (Rigby et al., 2016). Biosolids compost (COMP) and alkaline treated biosolids (ATB) will have lower TN contents due to the losses during processing and dilution through the addition of low-nitrogen agents during the composting or alkaline admixing process (Aubain et al., 2001). The N mineralization rate from COMP can also be affected by compost maturity and immobilization of N in compost-amended soil (Bowden et al., 2007; Doublet et al., 2010; Boen & Haraldsen, 2011). ATB has the lowest N concentrations, about 0.6% from twelve different biosolids (O'Connor et al., 2004). Christie et al. (2001) reported that ATB was suitable to increase soil pH, which has been further supported by Price et al. (2015). Although there have been many studies of the effects of the land application of biosolids on N bioavailability (Cogger et al., 2001; Mantovi et al., 2005; Spargo et al., 2008; Li et al., 2013), comparing plant growth and the availability of nitrogen from different sources is important to develop strategies to maintain soil fertility and improve crop production. Moreover, it is important to understand the effects of surface spreading and incorporated application in acidic soil over multiple years, on soil and plant nitrogen dynamics.

The objective of this study was to examine seasonal soil mineral nitrogen dynamics and plant nitrogen uptake after field application of three differently processed biosolids, with and without urea supplementation, and under two management practices (incorporation vs. surface spreading) over three consecutive years.

2.2 Materials and methods

2.2.1 Site description and treatments

A three-year field experiment was conducted using three biosolids (ATB, COMP, and LMAD) applied to plots at a research site located in Bible Hill, Nova Scotia, Canada (45.383910, -63.242706). The soil at this site has an acidic reddish-brown sandy loam texture and is mapped as an Ortho-humic Podzol as part of the Truro Association (CanSIS, 1991).



*SS: Surface spreading, INC: Incorporated

Fig. 2.1 The layout of the field experiment design

A 90 m x 38 m section of an agricultural research field was divided into four blocks, each block consisting of 15 plots. The plots measured 6 m in width x 8 m in length. The 15 treatments were randomized within each block. Treatments consisted of three types of biosolids (ATB, COMP, and LMAD), two application methods (surface spreading and incorporation), two biosolid application rates (full and a half), and one commercial fertilizer (urea). The detailed field plot application schedule for biosolids is shown in Table 2.1. Biosolid and urea were applied based on the recommended N requirement for corn (120 kg ha^{-1}) and the following assumptions of annual available N mineralized from the biosolids: ATB (50%), LMAD (75%), and COMP (50%). All the plots received ~30 kg N at planting with the corn seed, and the actual rate of N amendments added each year is shown in Table 2.2 and 2.3. The application rate was calculated based on the total N content of the biosolids (Table 2.4). Biosolids were weighed and spread manually to achieve a uniform distribution across each plot. Biosolids were incorporated to a depth of 15 cm using a tractor-mounted rototiller, and surface applied treatments were left alone after spreading. A 4m buffer zone was established in the center of the field, between Blocks 2 and 3, and 1 m buffer zones were established between the other blocks (Fig.2.1).

Table 2.1 Biosolids application schedule on the field

	2017		2018		2019
1st Soil sampling	5-May	1st Soil sampling	4-May	1st Soil sampling	29-Apr
Apply biosolids	10-May	Apply biosolids	16-May	Apply biosolids	7-May
Planting	25-May	Planting	24-May	Planting	31-May
urea sidedress	4-Jul	2nd Soil sampling	17-Jul	2nd Soil sampling	10-Jun
2nd Soil sampling	18-Jul	urea sidedress	17-Jul	3rd Soil sampling	8-Jul
3rd Soil sampling	7-Sep	3rd Soil sampling	14-Aug	4th Soil sampling	7-Aug
Harvest	17-Oct	4th Soil sampling	20-Sep	urea sidedress	7-Aug
4th Soil sampling	22-Nov	Harvest	15-Oct	5th Soil sampling	13-Sep
		5th Soil sampling	5-Nov	Harvest	22-Oct
				6th Soil sampling	30-Oct

Table 2.2 Application rates of biosolids and urea fertilizer added each year on the experimental plots (Full rate)

	ATB			**MAD**			**COMP**		
	2017	**2018**	**2019**	**2017**	**2018**	**2019**	**2017**	**2018**	**2019**
kg BS plot^{-1}	139.35	123.64	191.83	42.70	38.57	53.64	199.51	176.86	170.88
BS* (dry kg) plot^{-1}	85.81	76.46	119.19	9.77	8.96	9.34	84.49	73.85	76.46
kg BS ha^{-1}	29032.26	25758.74	39963.92	8895.48	8035.53	11174.34	41564.68	36844.97	35599.96
BS (dry kg) ha^{-1}	17878.06	15929.20	24831.58	2036.17	1866.25	1944.89	17602.64	15384.62	15929.20
Estimated TN BS (kg dry) ha^{-1}	172.52	180.00	180.03	144.57	120.00	120.00	239.40	180.00	180.00
TP BS (kg dry) ha^{-1}	119.25	105.93	145.26	50.61	63.42	75.98	156.08	130.62	101.71
TK BS (kg dry) ha^{-1}	141.06	135.08	27.31	7.90	12.12	15.65	42.01	30.92	25.33
Seed fertilizer*									
fert N kg ha^{-1}	36.00	38.00	30.00	36.00	38.00	30.00	36.00	38.00	30.00
fert P kg ha^{-1}	92.00	60.00	15.00	92.00	60.00	15.00	92.00	60.00	15.00
fert K kg ha^{-1}	0.00	6.00	27.00	0.00	6.00	27.00	0.00	6.00	27.00
TN applied kg ha^{-1}*	**208.52**	**218.00**	**210.03**	**180.57**	**158.00**	**150.00**	**275.40**	**218.00**	**210.00**
TP applied kg ha^{-1}	**211.25**	**165.93**	**160.26**	**142.61**	**123.42**	**90.98**	**248.08**	**190.62**	**116.71**
TK applied kg ha^{-1}	**141.06**	**141.08**	**54.31**	**7.90**	**18.12**	**42.65**	**42.01**	**36.92**	**52.33**

*BS: biosolids, Fert: fertilizer, Urea: applied 90 kg N ha^{-1} of full rate treatment.

*Actual TN was calculated on the amount of biosolids applied and not the available N which is based on the estimated mineralization rates

Table 2.3 Application rates of biosolids and urea fertilizer added each year on the experimental plots (Half rate)

	ATB			**MAD**			**COMP**		
	2017	**2018**	**2019**	**2017**	**2018**	**2019**	**2017**	**2018**	**2019**
kg BS plot^{-1}	69.68	61.82	95.91	21.35	19.29	26.82	99.76	88.43	85.44
BS* (dry kg) plot^{-1}	42.91	38.23	59.60	4.89	4.48	4.67	42.25	36.92	38.23
kg BS ha^{-1}	14516.13	12879.37	19981.96	4447.74	4017.77	5587.17	20782.34	18422.48	17799.98
BS (dry kg) ha^{-1}	8939.03	7964.60	12415.79	1018.09	933.13	972.45	8801.32	7692.31	7964.60
Estimated TN BS (kg dry) ha^{-1}	86.26	90.00	90.01	72.28	60.00	60.00	119.70	90.00	90.00
TP BS (kg dry) ha^{-1}	59.62	52.96	72.63	25.30	31.71	37.99	78.04	65.31	50.85
TK BS (kg dry) ha^{-1}	70.53	67.54	13.66	3.95	6.06	7.82	21.01	15.46	12.66
*UREA									
Fert urea kg plot^{-1}	0.47	0.47	0.47	0.47	0.47	0.47	0.47	0.47	0.47
Fert N kg plot^{-1}	0.22	0.22	0.22	0.22	0.22	0.22	0.22	0.22	0.22
Fert N kg ha-1	45.00	45.00	45.00	45.00	45.00	45.00	45.00	45.00	45.00
Seed fertilizer*									
Fert N kg ha^{-1}	36.00	38.00	30.00	36.00	38.00	30.00	36.00	38.00	30.00
Fert P kg ha^{-1}	92.00	60.00	15.00	92.00	60.00	15.00	92.00	60.00	15.00
Fert K kg ha^{-1}	0.00	6.00	27.00	0.00	6.00	27.00	0.00	6.00	27.00
TN applied kg ha^{-1}*	**167.26**	**173.00**	**165.01**	**153.28**	**143.00**	**135.00**	**200.70**	**173.00**	**165.00**
TP applied kg ha^{-1}	**151.62**	**112.96**	**87.63**	**117.30**	**91.71**	**52.99**	**170.04**	**125.31**	**65.85**
TK applied kg ha^{-1}	**70.53**	**73.54**	**40.66**	**3.95**	**12.06**	**34.82**	**21.01**	**21.46**	**39.66**

*BS: biosolids, Fert: fertilizer

*Actual TN was calculated on the amount of biosolids applied and not the available N which is based on the estimated mineralization rates

2.2.2 Biosolids

An alkaline treated biosolid (ATB) was obtained from the Halifax Biosolids Facility operated by N-VIRO Systems Canada Ltd., owned by the Walker Group, in Halifax, Nova Scotia, Canada. Biosolids compost was obtained from Fundy Compost Incorporated, Brookfield, Nova Scotia, which used Class B sewage solid mixed with woody residuals to generate a Class A compost. A liquid mesophilic anaerobically digested biosolid (LMAD) was obtained from a wastewater treatment plant in St. Hyacinthe, Quebec. The biosolid samples were collected in the field before applying and were sent to the Nova Scotia Department of Agriculture Analytical Laboratory (Bible Hill, Nova Scotia) for nutrient analysis. The properties of these three biosolids are presented in Table 2.4

Table 2.4 Selected characteristics of three types of biosolids (n=2)

	ATB			LMAD			COMP		
	2017	2018	2019	2017	2018	2019	2017	2018	2019
Dry Matter (%)	61.6	61.8	62.1	22.9	23.2	17.4	42.4	41.8	44.8
pH (pH Units)	10.0	9.8	8.7	7.8	8.2	8.1	7.4	7.4	7.0
Nitrogen (%)	1.0	1.1	0.7	5.6	6.4	6.2	1.4	1.2	1.1
Ammonium-N (%)	<0.01	0.1	0.1	1.3	1.3	1.3	0.0	0.1	0.1
Calcium (%)	16.5	16.5	22.9	1.9	2.8	3.8	2.0	1.4	1.2
Potassium (%)	0.8	0.9	0.1	0.4	0.7	0.8	0.2	0.2	0.2
K_2O (%)	1.0	1.0	0.1	0.5	0.8	1.0	0.3	0.2	0.2
Phosphorus (%)	0.7	0.7	0.6	2.5	3.4	3.9	0.9	0.9	0.6
P_2O_5 (%)	1.5	1.5	1.3	5.7	7.8	9.0	2.0	1.9	1.5
Magnesium (%)	0.3	0.3	0.1	0.3	0.4	0.5	0.2	0.2	0.2
Sodium (%)	0.1	0.1	0.0	0.2	0.3	0.4	0.1	0.1	0.0
Boron (ppm)	18.8	19.4	<10	13.7	12.1	12.0	10.9	10.7	12.3
Copper (ppm)	99.4	93.9	71.9	150.2	136.7	138.5	61.0	66.0	26.7
Iron (ppm)	7660	8129	3411	19806	35212	28122	6112	6551	6223
Manganese (ppm)	218.5	449.0	255.8	286.9	118.4	122.1	1172.1	1093.5	1114.4
Zinc (ppm)	207.6	248.1	178.1	212.1	238.0	280.8	175.4	198.3	123.8
C: N ratio	NA	30.9	38.5	NA	8.8	8.9	NA	38.6	37.9

2.2.3 Soil and plants sampling

Baseline sampling (Table 2.5) was conducted prior to biosolid applications, which included the collection of sixteen soil samples from random locations around the established field. Each composite soil sample consisted of ten soil cores taken from a 0 to 15 cm depth in a 3 m radius from each of the 16 areas around the field. Over the growing season, a composite soil sample was taken from each plot, consisting of ten cores collected from a depth of 0 to 15 cm, approximately monthly from May to November. The soil samples were air-dried and sieved to 2 mm, and the air-dried soils were bagged and stored at room temperature (20°C).

Table 2.5 Properties of baseline soil (0-15cm depth) in the experimental field (n=4)

	Soil
pH (pH Units)	5.63±0.21
Carbon (%)	1.13±0.10
Nitrogen (%)	0.20±0.02
Phosphorus (mg kg^{-1})	149.7±18.83
Potassium (mg kg^{-1})	133.95±25.81
Calcium (mg kg^{-1})	1318.73±128.94
Magnesium (mg kg^{-1})	165.95±20.15
Sodium (mg kg^{-1})	698.13±12.04
Sulphate (mg kg^{-1})	46.23±5.38
Aluminum (mg kg^{-1})	1557.98±28.37
Copper (mg kg^{-1})	3.53±0.62
Iron (mg kg^{-1})	188.55±16.83
Manganese (mg kg^{-1})	74.7±9.51
Zinc (mg kg^{-1})	1.78±0.28

*Values are presented as means±SD (n=4)

Whole plants were harvested from the middle two rows of each plot at the end of the growing season using a small plot corn harvester (International 484, Louisville, KY, USA). Two kraft paper bags of subsamples (silage) of the plant material were collected for each plot, weighing approximately 250 g of each bag, and oven-dried at 70 °C, ground using a Wiley mill (Thomas Scientific, Swedesboro, NJ, USA), and stored at 20°C until ready for analysis.

2.2.4 Laboratory analysis

Soil samples were extracted to determine soil inorganic N concentrations, i.e., nitrate-N (NO_3-N) and ammonium-N (NH_4-N), using 2M KCL at a 3:1 ratio of extractant to the soil. Extracted samples were analyzed using a Bran and Luebbe AutoAnalyzer III (Seal, Southampton, UK). Soil pH was determined in 0.01 mol L^{-1} $CaCl_2$ using a 2:1 ratio of $CaCl_2$ to the soil using a Fisher Scientific Accumet Excel XL50. All plant tissue samples were analyzed for total C (TC) and N (TN) using a LECO CNS-2000 analyzer (LECO, St. Joseph, MI).

2.2.5 Seasonal soil mineral nitrogen (SMN) dynamics

Soil mineral N (SMN) (NO_3-N and NH_4-N) was measured from all the soil samples collected at each sampling period over the three years. The data were converted into a soil nitrogen ratio (SNR), which is the ratio of the SMN of treated soil relative to the SMN of control soil, shown in Equation (1). SNR was used to represent the relative increase or decrease in the SMN pool based on the treatment applied.

$$SNR = \frac{SMN\ treatment}{SMN\ control} \qquad (1)$$

Soil mineral nitrogen exposure during the growing season due to biosolid types, rates, and management practices. Using the trapezoidal rule (Tallarida et al., 1987) was calculated beginning with the first sampling time in the given year (t=0) and ended with the last sampling time of the year. In this study, four sampling dates, at approximately the same time each year, were selected to calculate the area under the curve (Table 2.6) (Price et al., 2015),

$$\mathrm{AUC_{SMN}} = \frac{\sum[(SMN_{n-1}+SMN_n)\times(t_n-t_{n-1})]}{2} \qquad (2)$$

Table 2.6 Details of selected sampling dates for AUC calculation

Year	First	Second	Third	Forth
2017	4-May	18-Jul	7-Sep	22-Nov
2018	4-May	17-Jul	20-Sep	13-Nov
2019	29-Apr	8-Jul	13-Sep	20-Oct

2.2.6 Nutrient use efficiency (NUE)

Nutrient use efficiency (NUE) refers to the ability of a plant species to utilize nutrients to grow, and it can be expressed in several ways (Fageria & Baligar, 2001). In this study, NUE was described as an agronomic efficiency (AE), which is the increase in crop yield per unit nutrient applied (Baligar et al., 2001 and Roberts, 2008)). Estimated AE was presented as the increase in crop yield per unit of estimated nutrient availability, calculated as a yield increment from each treatment minus the control and divided by the recommended N requirement for corn (120 kg N ha^{-1}).

$$\text{AE} = \frac{Yield\ T - Yield\ C}{Actual\ amount\ of\ nitrogen\ applied} \qquad (3)$$

Where Yield T (kg ha^{-1}) refers to dry biomass yield of treatments, and Yield C (kg ha^{-1}) refers to dry biomass yield of the control, and the actual amount of nitrogen applied (kg N ha^{-1}) refers to total nitrogen applied from treatments each year (Table 2.2 and 2.3). The unit of AE is kg DM kg^{-1} N.

Apparent nutrient recovery efficiency (ANR) for nitrogen was used to indicate the ability of corn to absorb the applied nutrient from soil (Baligar et al., 2001). Estimated ANR was presented as the nutrient uptake of corn using the estimated nutrient availability in soil, calculated as the increment of nitrogen uptake from treatments divided by N requirement for corn (120 kg N ha^{-1}).

$$ANR = \frac{Plant\ total\ nutrient\ \ T - Plant\ total\ nutrient\ C}{Actual\ amount\ of\ nutrient\ applied} \times 100\% \quad (4)$$

Where Plant total nutrient T (kg ha^{-1}) refers to corn nitrogen uptake of from treatments plot, and Plant total nutrient C (kg ha^{-1}) refers to nitrogen uptake from the control plot, and the actual amount of nutrient applied (kg ha^{-1}) refers to total nitrogen applied from each treatment each year (Table 2.2 and 2.3).

2.2.7 Statistical analysis

Soil pH, soil mineral nitrogen (SMN), soil nitrogen ratio (SNR), plant total nitrogen (PTN), and corn yield were analyzed using a repeated-measures analysis of variance with the PROC

MIXED procedure in SAS v.9.4 (Statistical Analysis System version 9.4, SAS Institute, Raleigh, North Carolina). Analysis of variance was carried out within biosolid types. For each biosolid type, there were two factors of interest: amendment rate, management practice, which were examined over three years of study and are considered to be fixed effects, while the block is considered to be a random effect. Significant differences were based on an alpha value of 0.05. Multiple means comparisons, where necessary, were conducted using the Fisher Least Significant Difference test at an alpha value of 0.05. For each response, the assumption of normality was violated, a power transformation was applied to the original data, and the residuals were examined for normality (Montgomery, 2017). The results shown in the tables and figures were converted back to the original scale after analysis.

2.3 Results

2.3.1 Climatic conditions

The average monthly temperatures from 2017 to 2019 throughout the growing season are shown in Fig.2.2 (Environment Canada, 2020). The total monthly precipitation varied greatly in terms of timing and quantity during the three growing seasons (Fig. 2.3). The total precipitation in April and June 2018 was three times greater than the same period in 2017. The precipitation was high in 2019, especially in spring (April to June). Moreover, a hurricane passed through Nova Scotia in early September 2019, causing some damage to the corn plants in the experimental plots.

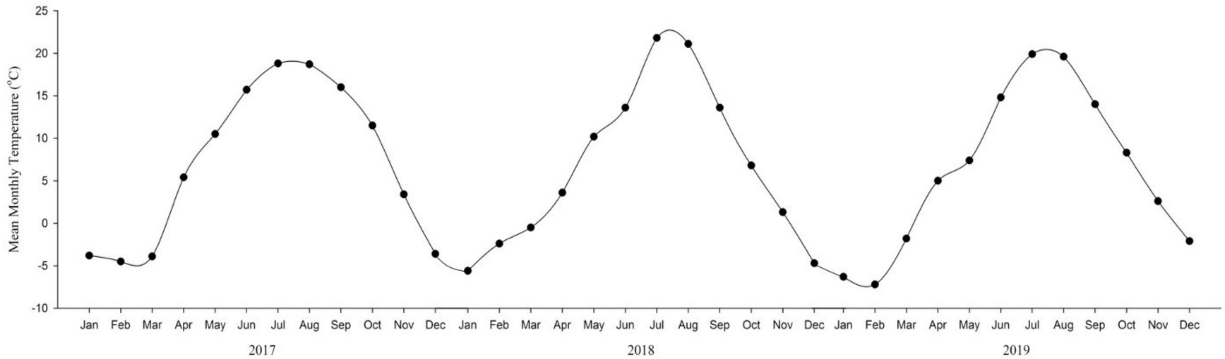


Fig. 2.2 Mean monthly temperature (°C) from 2017 to 2019 from Tatamagouche station, Nova Scotia (Environment Canada, 2020)

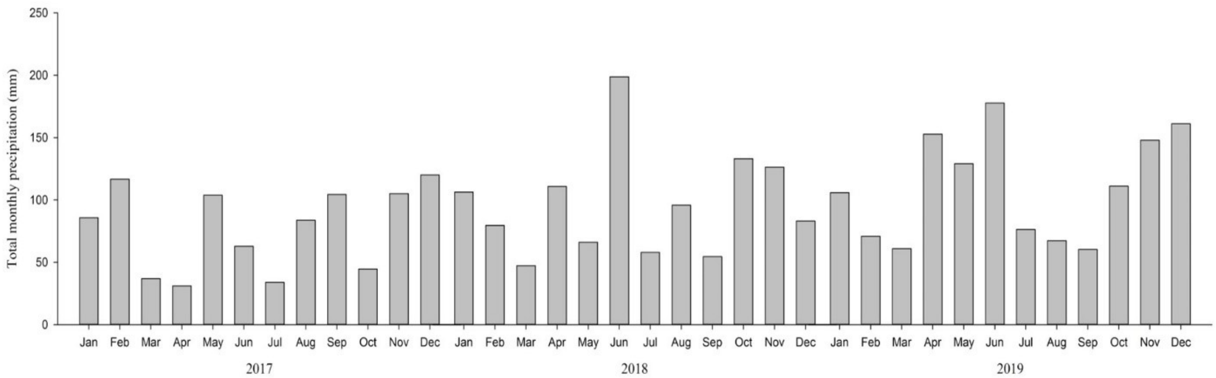


Fig. 2.3 Total monthly precipitation (mm) over the study period (2017-2019) from Tatamagouche station, Nova Scotia (Environment Canada, 2020)

2.3.2 Soil pH

In this study, highly significant two-way interaction effects between management practices (MP) x biosolids types and between year x biosolids types on soil pH were determined (Table 2.7). In both management practices, soil pH in ATB treatments was significantly higher than other treatments, and no difference was detected between COMP and the control treatment (Table 2.8). Soils receiving ATB treatments had significantly higher soil pH than the COMP, LMAD, control, and urea treatments (Table 2.9), reaching a maximum pH of 6.63. Soil pH in the COMP and control treatments in 2018 and 2019 were higher than those in 2017. No differences were detected among years for soil pH under urea treatments except in 2018. Moreover, no significant differences in soil pH were detected among LMAD treatments. Seasonal effects on soil pH of the different biosolid types over 2017 to 2019 are shown in Fig.2.4. A significantly increasing pH in the ATB treatments is apparent compared to other treatments, shifting from 5.5 to over 6.5 over the study period.

Table 2.7 ANOVA P-values for the main and interaction effects of management practice (MP), biosolid type, and sampling period (Year) on changes in soil pH from 2017 to 2019

	Soil pH
Biosolid	<.0001
MP	0.5396
Biosolid × MP	**0.0463**
Year	<.0001
Biosolid × Year	**<.0001**
MP × Year	0.9122
Biosolid × MP × Year	0.5418

*Significant effects that needed multiple means comparison are shown in bold.

Table 2.8 Effect of the two-way interaction of management practices x Biosolid types on soil pH over all rates from 2017 to 2019 (n=4)

MP	Biosolid types	Soil pH
Incorporation	Control	5.50 BC
	ATB	6.26 A
	COMP	5.53 B
	LMAD	5.38 D
	Urea	5.41 CD
Surface spreading	Control	5.50 BC
	ATB	6.20 A
	COMP	5.48 BC
	LMAD	5.42 CD
	Urea	5.57 B

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

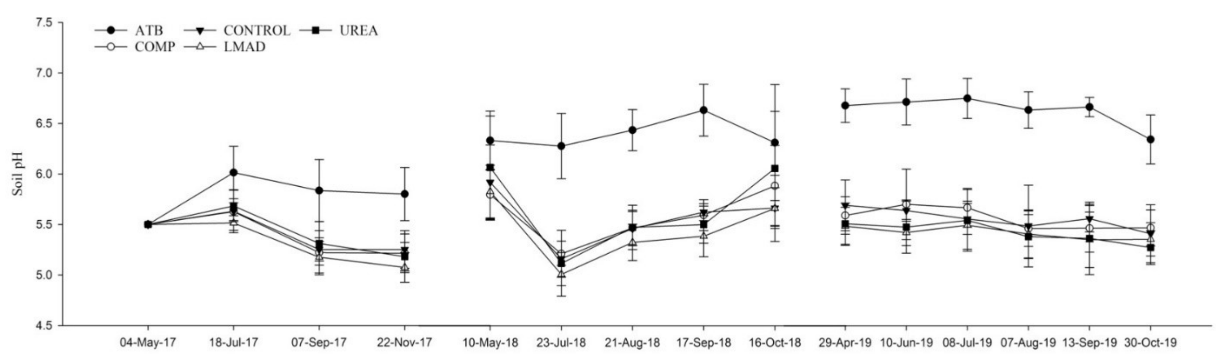


Fig. 2.4 Mean soil pH for unamended soil control, fertilizer Urea, three biosolids (ATB, COMP and LMAD) across three years (2017-2019)

Table 2.9 Effect of the two-way interaction of year x biosolid types on soil pH from 2017 to 2019 (n=4)

Biosolid types	Year	Soil pH
Control	2017	5.38 E
	2018	5.56 CD
	2019	5.56 CD
ATB	2017	5.67 C
	2018	6.40 B
	2019	6.63 A
COMP	2017	5.36 E
	2018	5.59 C
	2019	5.56 CD
LMAD	2017	5.34 E
	2018	5.44 E
	2019	5.42 E
Urea	2017	5.41 DE
	2018	5.64 C
	2019	5.42 E

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

2.3.3 Seasonal soil nitrogen dynamics and plant N uptake

A treatment rate of 120 kg available N ha^{-1} was used as the basis for all full rate treatments, and the biosolid applications were initially based on estimated mineralization rates for each of the biosolids types. The estimated mineralization rates were based on literature values but may not have necessarily represented the actual rates that these biosolids mineralized in the field. Therefore, the actual total N added for each biosolid type was higher than 120 kg N ha^{-1} (Table 2.2 and 2.3). On that basis, it was decided to compare response parameters for each treatment against the unamended soil control and urea treatments.

2.3.3.1 ATB amended soils

The ANOVA analysis (Table 2.10) shows that total soil mineral nitrogen (SMN), area under curve (AUC), and yield measured over the three years had significant two-way interaction effects of the rate of ATB x MP. Furthermore, plant total N (PTN), yield, agronomic efficiency (AE), and apparent nutrient recovery efficiency (ANR) displayed a significant two-way interaction of rate of ATB x year. AUC also had a significant two-way interaction effect of MP x year. SNR only had significant main effects on the rate of ATB, MP, and year but PTN only had significant main effects on the rate of ATB and MP. AE(N) and ANR (N) displayed a significant main effect of MP.

Table 2.10 ANOVA P-values for the main and interaction effects of management practice (MP), ATB, and sampling period (Year) on changes in total soil mineral nitrogen (SMN) (mg kg^{-1} soil), soil nitrogen ratio (SNR), area under curve (AUC) (mg SMN days kg $soil^{-1}$), plant total N (PTN) (kg N ha^{-1}), corn yield (kg DM ha^{-1}), agronomic efficiency (AE) (kg DM kg^{-1} N), and apparent nutrient recovery efficiency (ANR) from 2017 to 2019

	SMN	SNR	AUC	PTN	YIELD	AE (N)	ANR(N)
ATB	<.0001	**<.0001**	<.0001	**0.0003**	<.0001	0.011	0.3678
MP	0.0015	**0.0205**	0.0002	**0.0067**	<.0001	**<.0001**	**0.0002**
ATB × MP	**0.0079**	0.1321	**0.0182**	0.2434	**<.0001**	0.0559	0.8228
Year	0.0805	**0.0037**	<.0001	**0.002**	<.0001	<.0001	0.019
ATB × Year	0.7759	0.3035	0.9109	0.3626	**<.0001**	**<.0001**	**0.014**
MP × Year	0.1954	0.4397	**0.0015**	0.3587	0.7832	0.7297	0.3264
ATB × MP × Year	0.5242	0.8464	0.0548	0.5738	0.9503	0.8729	0.527

*Significant effects that needed multiple means comparison are shown in bold.

2.3.3.1.1 Soil Mineral Nitrogen Dynamics

Over the three growing seasons, a significant two-way interaction of rate of ATB x MP was detected for SMN concentrations in ATB amended soils (Table 2.11). The SMN concentrations measured across all ATB treatments were significantly different from the control except for the surface applied ATB+U treatment. Incorporated urea treatment was significantly higher compared to all other treatments. For surface spreading over the corresponding period, no significant differences were detected among ATB, ATB+U, and urea (Table 2.11). When comparing across the management practices, SMN concentrations measured after applying ATB and ATB+U in the incorporation method were not statistically different from the surface spreading of ATB and urea. The surface spreading of ATB+U was significantly different from the incorporation of ATB+U, resulting in lower SMN concentrations than other treatments but still higher than the control.

Table 2.11 Effect of the two-way interaction of management practice (MP) x amendment treatment (ATB, Urea and control) on total soil mineral nitrogen (SMN) (mg kg^{-1} soil) from 2017 to 2019 (n=4)

MP	Amendment treatment	SMN (mg kg^{-1} soil)
Incorporation	Control	20.77 D
	ATB	35.85 BC
	ATB+U	42.05 B
	Urea	61.12 A
Surface spreading	Control	20.77 D
	ATB	35.77 BC
	ATB+U	29.85 CD
	Urea	39.97 BC

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Peak SMN concentrations of incorporated treatments in the soil occurred in June or July over the three years because of high temperature, but peak SMN concentrations of surface applied was after the date of urea sidedress. The highest and lowest peak SMN concentrations were in the 2018 urea treatment and the 2019 control treatment, around 171 mg kg^{-1} soil (incorporated) and 19 mg kg^{-1} soil, respectively (Fig.2.5 (a) and (b)). In both management practices, a similar trend of SMN concentration was observed for the ATB treatment in 2017, as well as the ATB+U treatment. The peak SMN concentration of the surface applied ATB+U treatment decreased over the three years from 68 mg kg^{-1} soil in 2017 to 40 mg kg^{-1} soil by 2019 (Fig.2.5 (b)). In contrast, the SMN of the incorporated ATB+U treatment in the same period was steadily around 85 mg kg^{-1} soil (Fig.2.5 (a)).

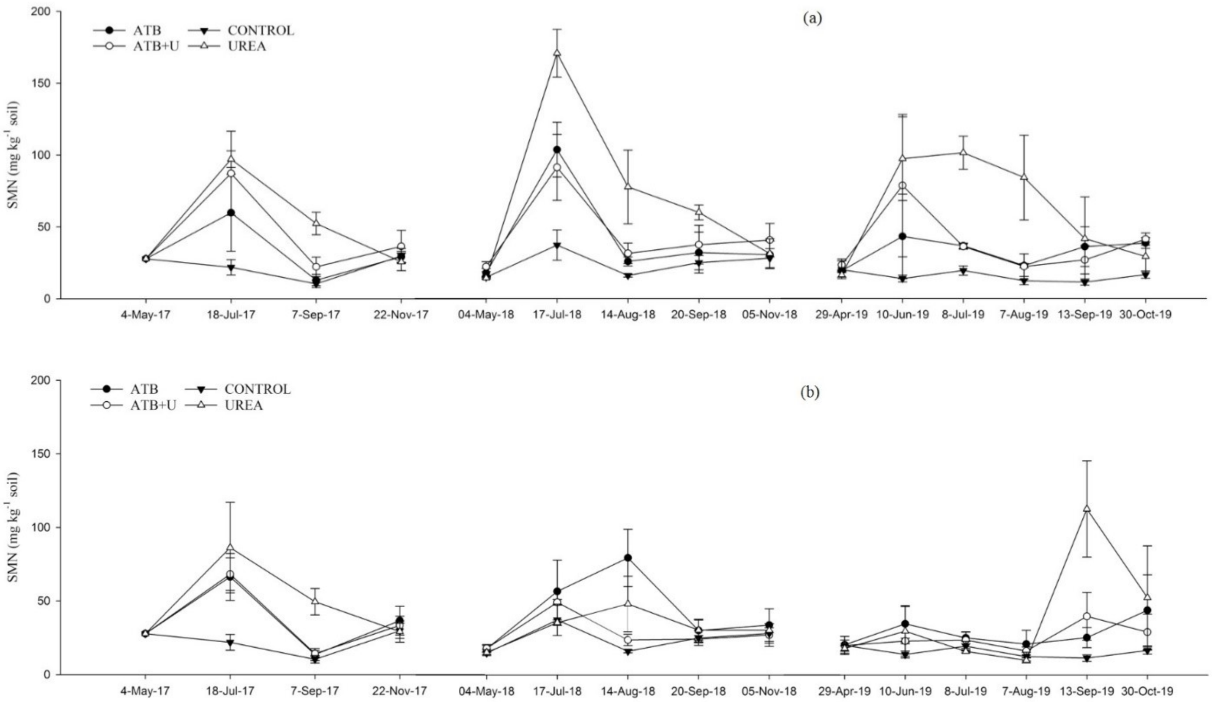


Fig. 2.5 Mean total soil mineral nitrogen (SMN) in mg kg^{-1} soil among ATB, ATB+U, control and urea application (a) within incorporated treatments across three years (b) within surface applied treatments across three years

The area under curve indicates total annual SMN exposure during each growing season. AUC values were generally higher for incorporation than surface spreading among urea, ATB, ATB+U, and urea treatments during the three growing seasons (Table 2.12). Moreover, AUC values for the ATB treatments and the ATB+U treatments were not significantly different in both management practices but were lower than those of the urea treatment. Across the management practices, AUC values for the incorporated treatments in 2018 were significantly higher than others (Table 2.13). No statistical differences in AUC values in both management practices were detected in 2017, as well as in 2019.

Table 2.12 Effect of the two-way interaction of management practice (MP) x amendment treatment (ATB, Urea and control) on the area under curve (AUC) (mg SMN days kg soil^{-1}) from 2017 to 2019 (n=4)

MP	Amendment treatment	AUC (mg SMN days kg soil^{-1})
Incorporation	Control	4201 D
	ATB	7746 BC
	ATB+U	8457 B
	Urea	13184 A
Surface spreading	Control	4201 D
	ATB	6463 BC
	ATB+U	6198 CD
	Urea	8371 B

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Table 2.13 Effect of the two-way interaction of management practice (MP) x year on the area under curve (AUC) (mg SMN days kg soil^{-1}) for the amendment treatment (ATB, ATB+U, urea, and control) (n=4)

MP	Year	AUC (mg SMN days kg soil^{-1})
Incorporation	2017	7894 B
	2018	11236 A
	2019	6061 BC
Surface spreading	2017	7443 B
	2018	6269 BC
	2019	5213 C

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

2.3.3.1.2 Soil nitrogen ratio

The soil nitrogen ratio (SNR) is a measure of the effect of the biosolid treatments relative to the control in a given year. The SNR in the incorporated application (2.39) was significantly higher than that in surface spreading (1.91). Urea provided almost three times the SMN concentrations in soil relative to the control, while the ATB and ATB+U treatments were almost two times greater (Table 2.14).

Table 2.14 Mean soil nitrogen ratio (SNR) conducted from amendment treatment (ATB, ATB+U, and urea) over both management practices from 2017 to 2019 (n= 4)

Amendment treatment	SNR
ATB	1.81 B
ATB+U	1.83 B
Urea	2.82 A

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different

Under both management practices, the SNR values in 2017 and 2018 returned to background levels by the end of the growing season, but all treatments in 2019 were over 2.5 times greater than those of the control (Fig. 2.6 (a) and (b)). In the incorporation treatments, urea provided the highest SMN over the three years, followed by ATB+U and ATB (Fig. 2.6 (a)). Moreover, the SNR value of the urea treatments had two peak points, in June (7) and August (6.8), as did the ATB treatments. A similar trend was observed for SNR in 2017 for the ATB and ATB+U treatments. However, the ATB treatment provided the highest SMN (5) in the 2018 surface applied method, followed by urea and ATB+U treatments (Fig. 2.6 (b)). The SNR values of the surface-applied urea treatment were around ten times greater than those of the control in September 2019

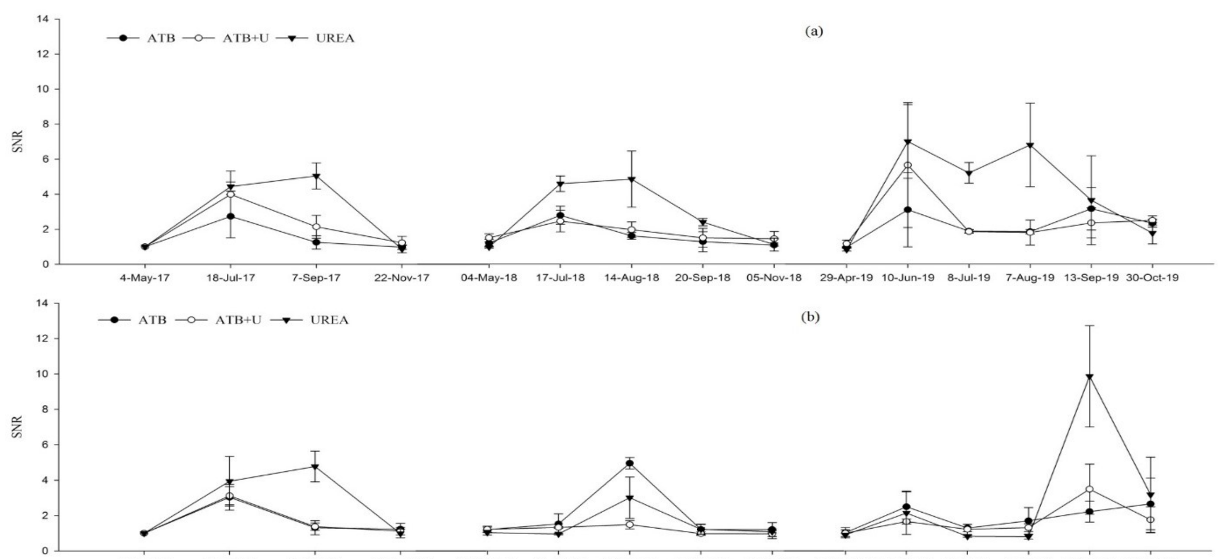


Fig. 2.6 Mean soil nitrogen ratio (SNR) in ATB, ATB+U, control and urea application across three years (a) within incorporated treatments and (b) within surface applied treatments

2.3.3.1.3 Crop yield

The whole-plant corn biomass yields measured under incorporated ATB and ATB+U treatments were not statistically different from each other. However, they were significantly higher than those of the rest of the treatments under both management practices (Table 2.15). The biomass for the incorporated ATB was 41% greater than control and urea, while incorporated ATB+U was 50% greater than those of the control. Incorporated urea treatment resulted in corn yield that was significantly different from yields of ATB in surface spreading. Concerning surface application, no statistical differences in yield were detected among the ATB, ATB+U, and urea treatments, but they were 15%, 13%, and 7% higher than the control, respectively.

Table 2.15 Effect of the two-way interaction of management practice (MP) x amendment treatment (ATB, Urea and control) on total crop yield (kg DM ha^{-1}) from 2017 to 2019 (n=4)

MP	Amendment treatment	Yield (kg DM ha^{-1})
Incorporation	Control	11980 D
	ATB	16901 A
	ATB+U	17940 A
	Urea	14848 B
Surface spreading	Control	11980 D
	ATB	13885 BC
	ATB+U	13488 C
	Urea	12805 CD

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

The corn yields measured under the ATB, ATB+U, and urea treatments showed a decreasing trend over the three years (Table 2.16). The ATB treatments were gradually greater than urea treatments from 1.5% to 37.8% over the three years, as well as ATB+U treatments (2% to 37%). The corn yields from ATB and ATB+U treatments in 2017 were significantly higher than the rest of the treatments but were not different from each other. In 2018, no differences were detected in corn yield among ATB, ATB+U, and urea treatments, but all were significantly higher than the control. The corn yield of the control treatment in 2017 was not statistically different from ATB, ATB+U, and control treatments in 2019

Table 2.16 Effect of the two-way interaction of the year x amendment treatments (ATB, Urea and control) on crop yield (kg DM ha^{-1}) over both management practices (n=4)

Amendment treatment	Year	Yield (kg DM ha^{-1})
Control	2017	13026 E
	2018	10539 F
	2019	12375 E
ATB	2017	17377 AB
	2018	15452 C
	2019	13351 DE
ATB+U	2017	18886 A
	2018	14977 C
	2019	13279 DE
Urea	2017	17128 B
	2018	14665 CD
	2019	9688 F

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Agronomic efficiency (AE) expresses the yield relative to the amount N added. The AE is possibly skewed because of the high denominator for ATB treatments as a total N application, so that these values may be underestimated for ATB and ATB+U treatments. The highest AE from 2017 to 2019 were incorporated ATB+U (48.69 kg DM kg^{-1} N), urea (40.85 kg DM kg^{-1} N) and ATB+U (19.45 kg DM kg^{-1} N), respectively (Fig. 2.7). Within each treatment, the AE showed a declining trend over the three years except ATB (Table 2.17). The AE of the incorporated application (26.91 kg DM kg^{-1} N) over the three years was significantly higher than surface application (7.89 kg DM kg^{-1} N), which was 53% higher in 2017, 41% in 2018, and 64% in 2019. The AE of ATB+U treatment was higher than ATB treatment over the three years, except in 2018 (Fig. 2.7). Estimated AE was based on the yield relative to an assumed idealized mineralization to achieve the 120 kg N ha^{-1}. The AE showed a positive linear relationship with estimated AE under both management practices from 2017 to 2019 (R^2=0.9705) with a slope of 1.5059 (Fig. 2.8), which means 0.66 kg DM kg^{-1} N from the actual AE would be required to increase the estimated AE by 1.0 kg DM kg^{-1} N, and the estimated AE was 1.5 times higher than the actual AE

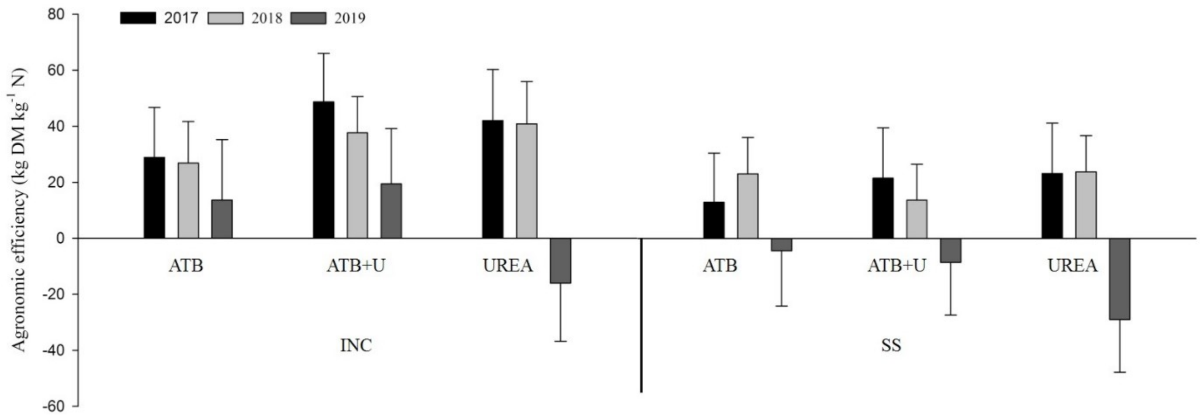


Fig. 2.7 Agronomic efficiency (kg DM kg^{-1} N) of ATB and urea treatments in both management practices over the three years

Table 2.17 Effect of the two-way interaction of the year x amendment treatments (ATB, Urea and control) on agronomic efficiency (AE) (kg DM kg^{-1} N) over both management practices (n=4)

Amendment treatment	Year	AE (kg DM kg^{-1} N)
ATB	2017	20.86 C
	2018	22.53 C
	2019	4.65 D
ATB+U	2017	35.04 A
	2018	25.65 BC
	2019	5.47 D
Urea	2017	32.56 AB
	2018	32.23 AB
	2019	-22.40 E

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

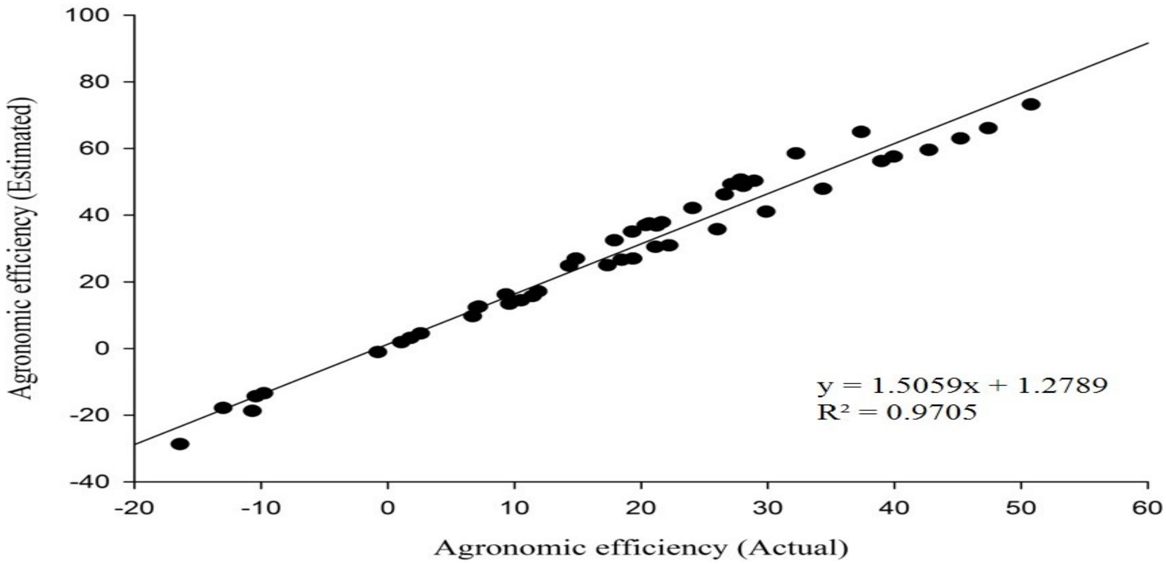


Fig. 2.8 The relationship between observed and estimated agronomic efficiency (kg DM kg^{-1} N) for ATB treatments under both management practices from 2017 to 2019

2.3.3.1.4 Plant uptake nitrogen

The total plant nitrogen (PTN) was expressed as the amount of nitrogen uptake from the soil. The PTN measured in the incorporated application (148.38 kg N ha^{-1}) was significantly higher than that in surface spreading (109.94 kg N ha^{-1}). No significant differences were observed among ATB, ATB+U, and urea treatments but were significantly higher than the control (Table 2.18).

Table 2.18 Mean plant total nitrogen (PTN) for the amendment treatments (ATB, ATB+U, urea, and control) over both management practices from 2017 to 2019 (n=4)

Amendment treatment	PTN (kg N ha^{-1})
Control	79.34 B
ATB+U	151.11 A
ATB	160.89 A
Urea	125.29 A

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Apparent nutrient recovery (ANR)(N) was N uptake relative to the amount of N added. The ANR (N) of the incorporated application (57.08%) over the three years was significantly higher than the surface application (27.01%). Under incorporated application, the ANR(N) measured on the ATB and ATB+U treatments had similar trends, which decreased in the first two years from 103.83% to 30.25% and 73.73% to 40.74%, respectively, then increased again in 2019, reached up to 31.71% and 68.98% (Fig. 2.9). However, the ANR(N) of the surface applied ATB treatments had an opposite trend, increasing to 30.04% in 2018 and declining to 23.15% in 2019. Moreover, the ANR(N) on the ATB treatments showed a decreasing trend over the three years (Table 2.19). No statistical difference was detected between the ATB and ATB+U treatments on ANR (N) over the three years. Estimated ANR(N) was N uptake relative to the assumed idealized available N (120 kg N ha^{-1}). The ANR(N) of urea treatments in 2018 was significantly higher than that in 2019. The ANR(N) showed a positive linear relationship with estimated ANR (N) under both management practices from 2017 to 2019 (R^2=0.9227) with a slope of 1.2883 (Fig. 2.10) which means 0.78 % from the actual ANR(N) would be required to increase the estimated ANR(N) by 1%, and the estimated ANR(N) was 1.3 times higher than the actual ANR(N).

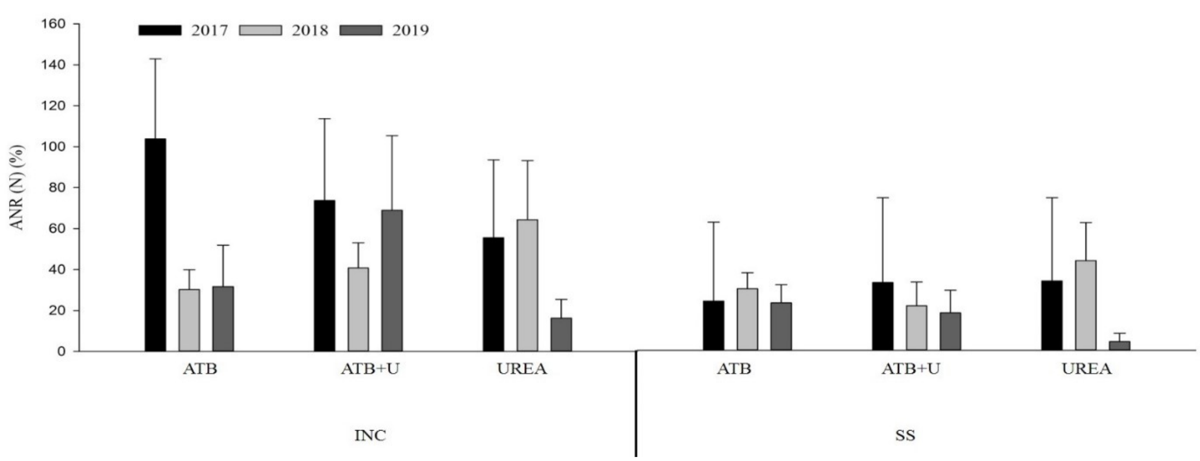


Fig. 2.9 Apparent nutrient recovery efficiency (ANR) of ATB and urea treatments in both management practices over the three years.

Table 2.19 Effect of the two-way interaction of the year x amendment treatments (ATB, Urea and control) on apparent nutrient recovery efficiency (ANR) (N) over both management practices (n=4)

Amendment treatment	Year	ANR (N)
ATB	2017	54.44 AB
	2018	27.04 BC
	2019	24.95 BC
ATB+U	2017	51.44 AB
	2018	31.24 BC
	2019	43.60 BC
Urea	2017	55.12 AB
	2018	76.89 A
	2019	13.66 C

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

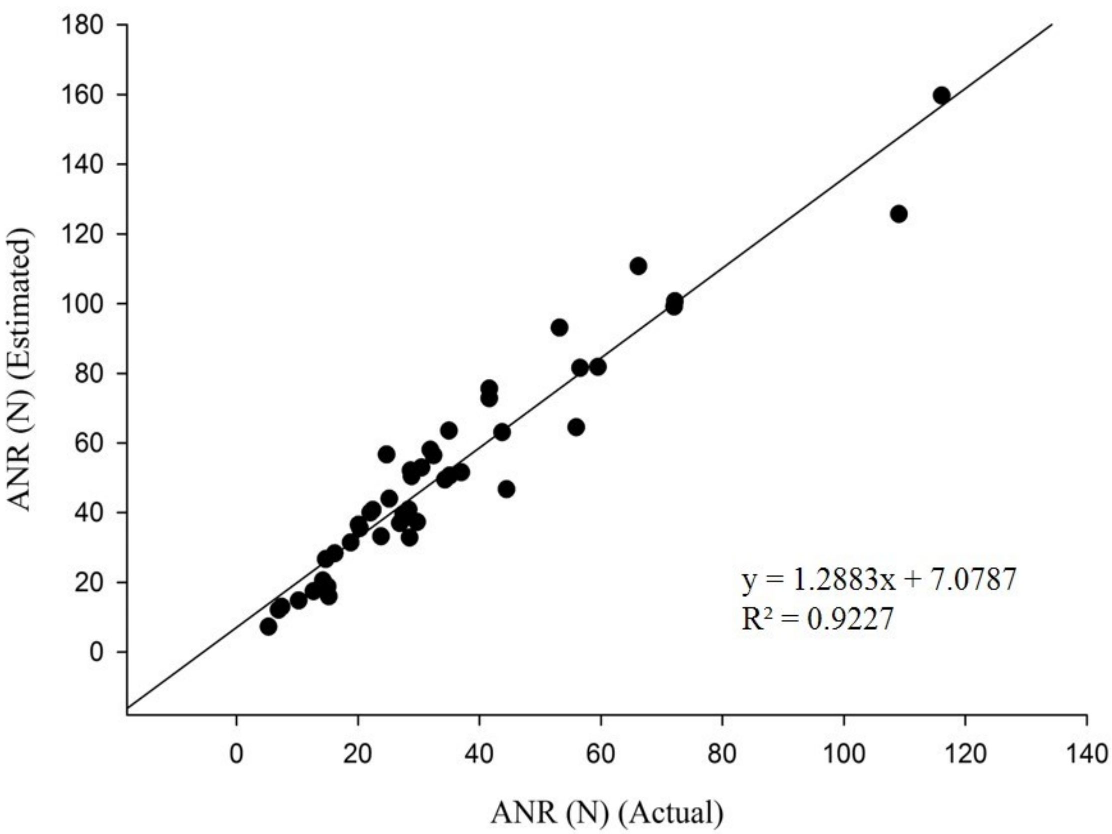


Fig. 2.10 The relationship between observed and estimated apparent nutrient recovery efficiency (N) for ATB treatments under both management practices from 2017 to 2019.

2.3.3.2 COMP amended soils

The ANOVA analysis (Table 2.20) indicates that a three-way interaction of the rate of COMP x MP x year was detected on AUC values for SMN. PTN, corn yield, AE (N), and ANR(N), measured over the three years, all had significant interaction effects of the rate of COMP x year. SMN and corn yield were affected by the two-way interaction of the rate of COMP x MP. PTN, AE (N), and ANR (N) measured over the three years all also displayed significant main effects of MP. SNR had significant main effects on the rate of COMP, MP, and year.

Table 2.20 ANOVA P-values for the main and interaction effects of management practice (MP), amendment treatment (COMP), and sampling period (Year) on changes in total soil mineral nitrogen (SMN) (mg kg^{-1} soil), soil nitrogen ratio (SNR), area under curve (AUC) (mg SMN days kg $soil^{-1}$), plant total N (PTN) (kg N ha^{-1}), corn yield (kg DM ha^{-1}), agronomic efficiency (AE) (kg DM kg^{-1} N), and apparent nutrient recovery efficiency (ANR) from 2017 to 2019

	SMN	SNR	AUC	PTN	YIELD	AE (N)	ANR(N)
COMP	<.0001	**<.0001**	<.0001	<.0001	0.0002	0.0004	<.0001
MP	0.0019	**0.0164**	0.0005	**0.0008**	<.0001	**<.0001**	**0.0003**
COMP× MP	**0.0107**	0.1932	0.0054	0.2503	**0.0122**	0.4843	0.2852
Year	0.3014	**0.0044**	<.0001	<.0001	<.0001	<.0001	<.0001
COMP × Year	0.9385	0.3689	0.8269	**0.0005**	**<.0001**	**<.0001**	**<.0001**
MP × Year	0.1289	0.2486	0.0032	0.7336	0.8611	0.3751	0.921
COMP × MP × Year	0.376	0.7259	**0.0114**	0.9699	0.9768	0.3663	0.6946

*Significant effects that needed multiple means comparison are shown in bold.

2.3.3.2.1 Soil Mineral Nitrogen Dynamics

No difference was detected in the SMN concentrations measured between the COMP treatments and the control in both MP. Urea was statistically higher than the COMP+U treatments in the incorporated method and exceeded 24 mg kg^{-1} soil, reaching 61.12 mg kg^{-1} soil (Table 2.21). Under the surface spreading treatments, only urea had significantly higher SMN concentrations. Across the management practices, incorporated application resulted in higher SMN than surface spreading application (Table 2.21).

Table 2.21 Effect of the two-way interaction of management practice (MP) and amendment treatment (COMP, Urea, and control) on total soil mineral nitrogen (SMN) (mg kg^{-1} soil) from 2017 to 2019 (n=4)

MP	Amendment treatment	SMN (mg kg^{-1} soil)
Incorporation	Control	20.77 C
	COMP	22.02 C
	COMP+U	37.30 B
	Urea	61.12 A
Surface spreading	Control	20.77 C
	COMP	21.74 C
	COMP+U	26.32 C
	Urea	39.93 B

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Peak SMN concentration of the COMP+U treatments in both management practices was higher than the COMP treatments and control over the three years. The peak SMN concentration of the incorporated COMP treatments increased over the three years from 30 mg kg^{-1} soil in 2017 to 57 mg kg^{-1} soil by 2019 (Fig.2.11 (a)). The surface applied COMP treatments also slightly increased from 30 mg kg^{-1} soil to 37 mg kg^{-1} soil (Fig.2.11 (b)). Averaged in each annual sampling, SMN in the COMP+U treatments was around 50% higher than that in the COMP treatments from 2017 to 2019, except surface spreading in 2018. The highest SMN of surface-applied urea treatments in 2018 was 50 mg kg^{-1} in August, while rest treatments were around 40 mg kg^{-1} in July.

Area under curve values (AUC) of incorporation was higher than surface spreading among urea, COMP, and COMP+U treatments during three growing seasons (Table 2.22). Comparing the AUC values of amendment treatments for incorporated applications across the years, all treatments showed the same trends over the three years, which increased in the first two years then went down in 2019. AUC values increased, over all three years, as the available N supply increased from the incorporated treatments, Urea > COMP+U > COMP > control. In contrast, in surface spreading treatments, this only was observed in 2017. Some significant differences within treatments but across years were also observed. In surface spread treatments, no significant differences between treatments were measured in 2018 and only for urea in 2019.

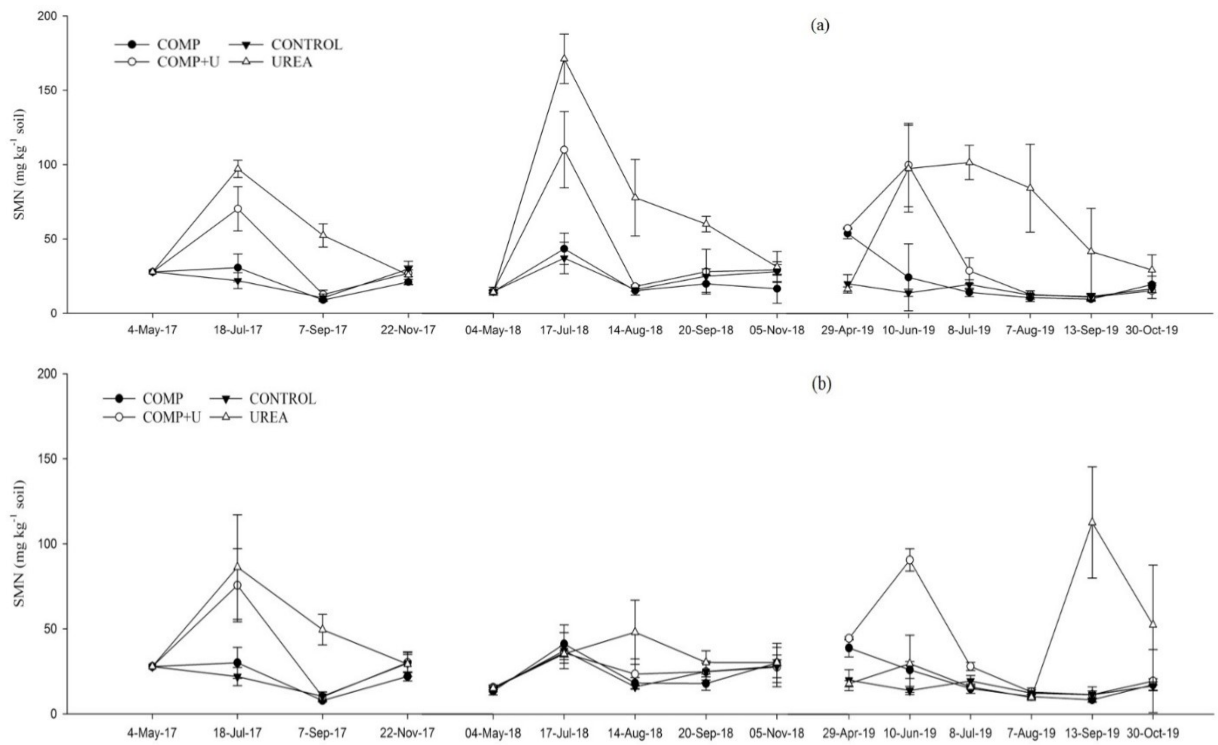


Fig. 2.11 Mean total soil mineral nitrogen (SMN) (mg kg^{-1} soil) among COMP, COMP+U, control and urea application across three years (a) within incorporated treatments and (b) within surface applied treatments

Table 2.22 Effect of the three-way interaction of management practice (MP), amendment treatment (COMP, Urea, and control), and year on area under curve (AUC) (mg SMN days kg $soil^{-1}$) (n=4)

MP	Amendment treatment	Year		
		2017	2018	2019
Incorporation	Control	4190 FG	5503 EFG	2911 G
	COMP	4305 FG	5204 EFG	3680 G
	COMP+U	7239 DEF	10824 BC	4776 FG
	Urea	11405 B	17978 A	10169 BCD
Surface spreading	Control	4190 FG	5503 EFG	2911 G
	COMP	4240 FG	5176 EFG	2649 G
	COMP+U	7537 CDEF	5395 EFG	2911 G
	Urea	10682 BCD	5910 EFG	8522 BCDE

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

2.3.3.2.2 Soil nitrogen ratio

The SNR, over all treatments, in the incorporated application (2.06) was significantly higher than that in the surface spreading (1.57). Urea provided almost three times SMN concentrations in the soil relative to the control, and the COMP+U treatments were 1.5 times greater than the control (Table 2.23).

Table 2.23 Mean soil nitrogen ratio (SNR) conducted from amendment treatment (COMP, COMP+U, and urea) from 2017 to 2019 (n=4)

Amendment treatment	SNR
COMP	1.07 B
COMP+U	1.56 B
Urea	2.82 A

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

In both management practices, the SNR values in 2017 and 2018 returned to background soil level by the end of the growing season, but the COMP and COMP+U treatments in 2019 were around 2.5 times greater than those of the control at the start of the growing season (Fig. 2.12 (a) and (b)). Urea treatments provided the highest SMN over the three years, followed by the COMP+U and COMP treatments. Similar patterns of SNR in 2017 and 2018 were observed for incorporated treatments. In 2019, the SNR values of the incorporated COMP+U treatments resulted in around seven times greater than that of the control in June (Fig. 2.12 (a)), as well as in surface spreading (Fig. 2.12 (b)). There is a bimodal shape in 2019 for urea treatments in both MP, occurring in June (7) and August (6.8) for incorporation and June (2.1) and September (9.9) for surface spreading.

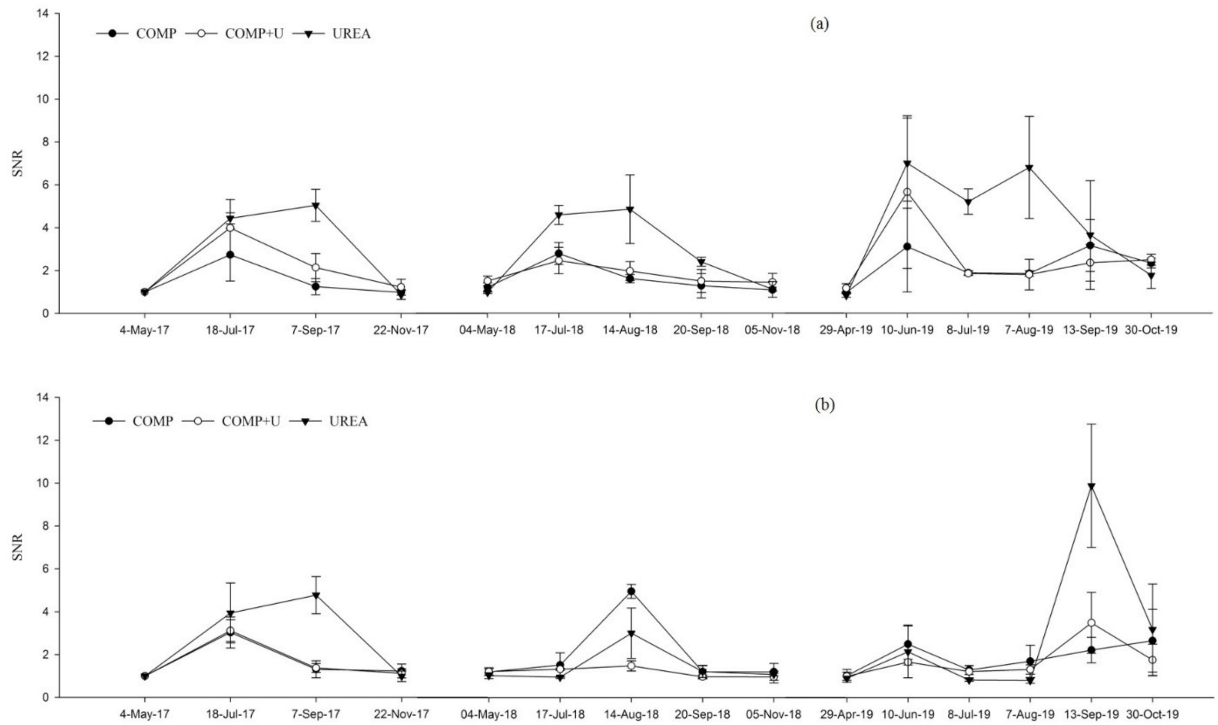


Fig. 2.12 Mean soil nitrogen ratio (SNR) in COMP, COMP+U, control and urea application across three years (a) within incorporated treatments and (b) within surface applied treatments

2.3.3.2.3 Crop yield

The whole-plant corn biomass yields measured under incorporated COMP+U and urea treatments were not statistically different from each other but were significantly higher than the rest of the treatments under both management practices (Table 2.24). The incorporated COMP treatments resulted in corn yields that were not different from the yield of urea in both management practices. Under surface spreading, biomass yields measured of the COMP+U treatments were 15% higher than the COMP but were not statistically different from urea and control treatments (Table 2.24).

Table 2.24 Effect of the two-way interaction of management practice (MP) and amendment treatment (COMP, Urea, and control) on crop yield (kg DM ha^{-1}) from 2017 to 2019 (n=4)

MP	Amendment treatment	Yield (kg DM ha^{-1})
Incorporation	Control	11980 DE
	COMP	14091 BC
	COMP+U	15724 A
	Urea	14848 AB
Surface spreading	Control	11980 DE
	COMP	10972 E
	COMP+U	12573 D
	Urea	12805 CD

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

A significant amendment treatment × year interaction effect on crop yield was shown in Table 2.25. The corn yield measured under the COMP, COMP+U, and urea treatment showed a decreasing trend over the three years. The COMP treatments were gradually greater than urea treatments from -12.8% to 20.2% over the three years, as well as COMP+U treatments (2.5% to 10.7%). The corn yield of COMP and urea treatments in 2017 was significantly higher than the rest of the treatment among years but was no different from each other. No significant difference was detected among crop yield measured with the COMP in 2017 and the COMP+U in 2018 but was significantly higher than all treatments in 2019 except the control. In 2019, the urea application resulted in corn yield that was significantly lower than the other three treatments except for the COMP+U.

The highest agronomic efficiency (AE) from 2017 to 2019 was determined for the incorporated urea (42.07 kg DM kg^{-1} N), urea (40.85 kg DM kg^{-1} N), and COMP (-0.57 kg DM kg^{-1} N), respectively (Fig. 2.13). Within each treatment, the AE measured showed a declining trend over the three years (Table 2.26). The AE of the incorporated application (21.77 kg DM kg^{-1} N) over the three years was significantly higher than surface application (2.09 kg DM kg^{-1} N), and was approximately 67% higher in 2017, 55% in 2018, and 800% in 2019. The AE of surface applied COMP treatment was below 0 over the three years (Fig. 2.13). The AE showed a positive linear relationship with estimated AE under both management practices from 2017 to 2019 (R^2=0.9634) with a slope of 1.2609 (Fig. 2.14), which means 0.79 kg DM kg^{-1} N from the actual AE would be required to increase the estimated AE by 1.0 kg DM kg^{-1} N, and the estimated AE was 1.3 times higher than the actual AE.

Table 2.25 Effect of the two-way interaction of year and amendment treatment (COMP, Urea, and control) on crop yield (kg DM ha^{-1}) over both management practices (n=4)

Amendment treatment	Year	Yield (kg DM ha^{-1})
Control	2017	13026 CDE
	2018	10539 GH
	2019	12375 DEF
COMP	2017	14925 B
	2018	11028 FGH
	2019	11641 EFG
COMP+U	2017	17562 A
	2018	14159 BCD
	2019	10724 FGH
Urea	2017	17128 A
	2018	14665 BC
	2019	9688 H

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

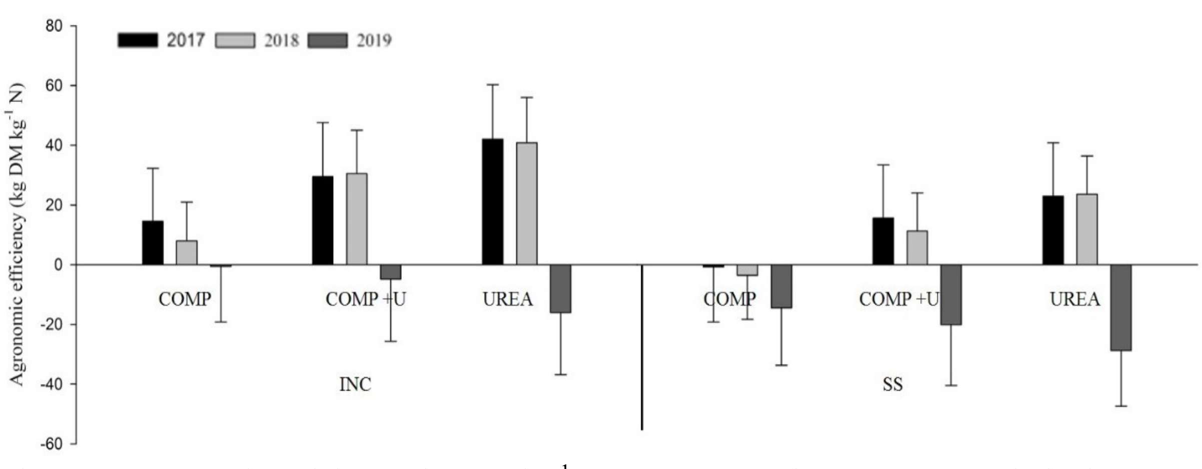


Fig. 2.13 Agronomic efficiency (kg DM kg^{-1} N) of COMP and urea treatments in both management practices over the three years

Table 2.26 Effect of the two-way interaction of year and amendment treatment (COMP, Urea, and control) on agronomic efficiency (AE) (kg DM kg^{-1} N) over both management practices (n=4)

Amendment treatment	Year	AE (kg DM kg^{-1} N)
COMP	2017	14.69 B
	2018	2.24 C
	2019	-3.50 C
COMP+U	2017	35.54 A
	2018	20.93 B
	2019	-4.92 C
Urea	2017	32.56 A
	2018	32.23 A
	2019	-22.40 D

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

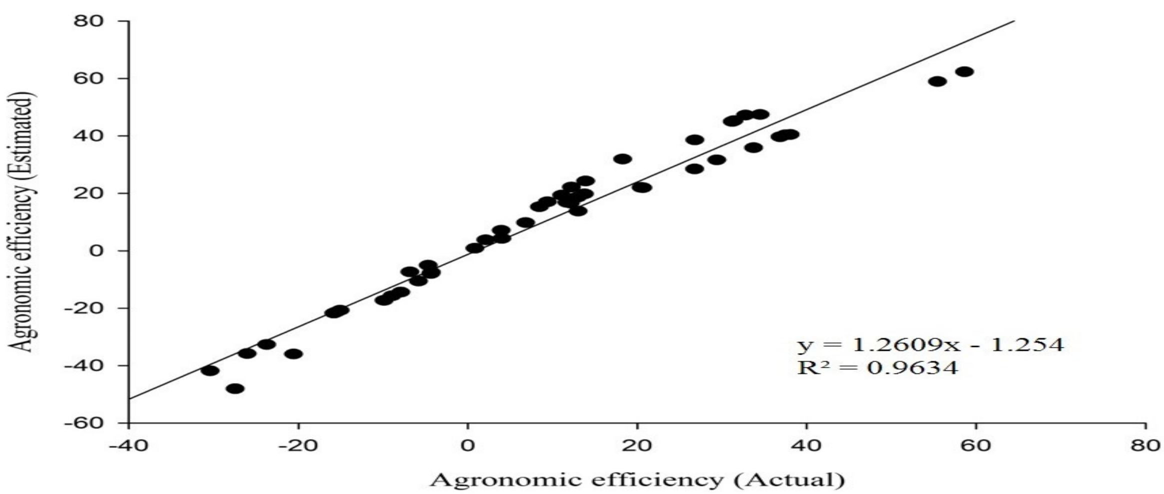


Fig. 2.14 The relationship between observed and estimated agronomic efficiency (kg DM kg-1 N) for COMP treatments under both management practices from 2017 to 2019.

2.3.3.2.4 Plant nitrogen uptake

The PTN in the incorporated application (109.68 kg N ha^{-1}) was significantly higher than that in surface spreading (94.46 kg N ha^{-1}). The PTN measured under the COMP+U and urea treatments in 2017 were significantly higher than all treatments across three years but were no different from each other (Table 2.27). The PTN of the COMP treatments was significantly lower than that of the COMP+U treatments over the three years except in 2019. In 2019, no statistical differences were observed among all treatments.

Table 2.27 Effect of the two-way interaction of year and amendment treatment (COMP, Urea, and control) on plant total nitrogen (PTN) (kg N ha^{-1}) over both management practices (n=4)

Amendment treatment	Year	PTN (kg N ha^{-1})
Control	2017	94.64 E
	2018	59.86 F
	2019	83.52 E
COMP	2017	117.09 CD
	2018	60.89 F
	2019	90.73 E
COMP+U	2017	149.90 AB
	2018	97.63 DE
	2019	94.66 E
Urea	2017	151.00 A
	2018	129.07 BC
	2019	95.81 E

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Apparent nutrient recovery (ANR) of the incorporated application (31.77%) over the three years was higher than surface application (15.91%). The ANR on the COMP+U of incorporation and surface spreading had a decreasing trend over the three years, starting at 49.55% to 12.63% and 37.05% to 0.87%, respectively (Fig 2.15). The ANR of the surface applied COMP treatments were all under 10% over the three years, as well as the same treatment of incorporation in the last two years. No statistical differences were detected for the ANR (N) of the COMP or COMP+U treatments over the three years (Table 2.). The ANR (N) showed a positive linear relationship with estimated ANR (N) under both management practices from 2017 to 2019 (R^2=0.9725) with a slope of 1.489 (Fig. 2.16) which means 0.67 % from the actual ANR(N) would be required to increase the estimated ANR(N) by 1%, and the estimated ANR(N) was 1.5 times higher than the actual ANR(N).

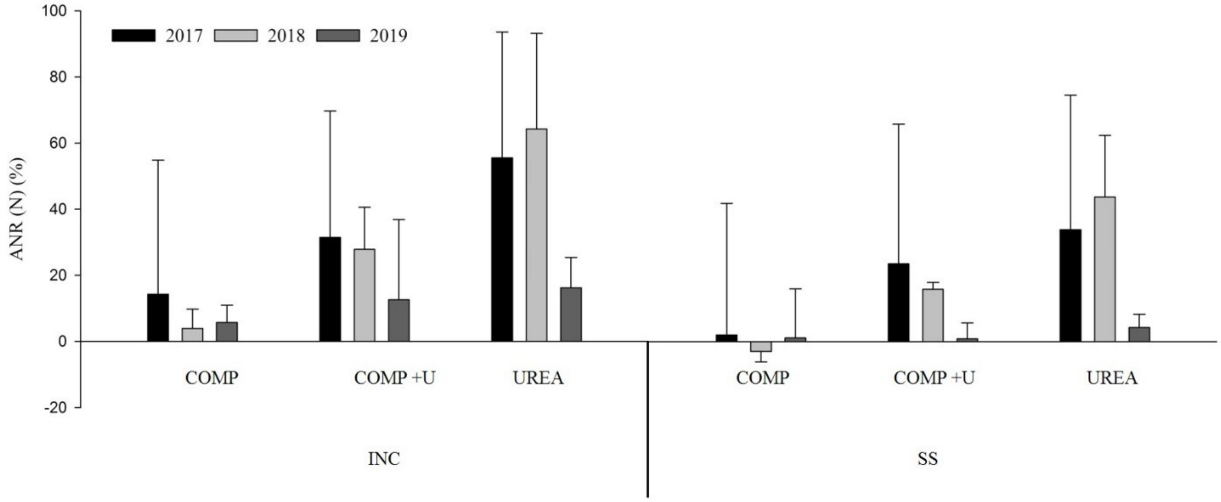


Fig. 2.15 Apparent nutrient recovery efficiency (ANR) of COMP and urea treatments in both management practices over the three years.

Table 2.28 Effect of the two-way interaction of year and amendment treatment (COMP, Urea, and control) on apparent nutrient recovery efficiency (ANR) (N) over both management practices (n=4)

Amendment treatment	Year	ANR (N)
COMP	2017	6.38 DE
	2018	0.47 E
	2019	3.43 E
COMP+U	2017	25.62 C
	2018	21.83 CD
	2019	11.14 CDE
Urea	2017	55.12 B
	2018	76.89 A
	2019	13.66 CDE

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

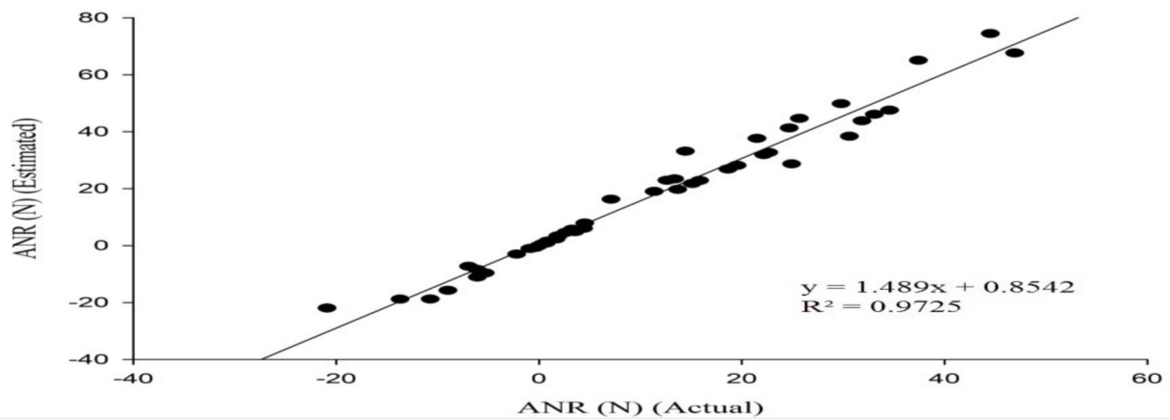


Fig. 2.16 The relationship between observed and estimated apparent nutrient recovery efficiency (N) for the COMP treatments under both management practices from 2017 to 2019.

2.3.3.3 LMAD amended soils

The statistical analysis (Table 2.29) illustrates that AUC value was profoundly affected by a three-way interaction of rate of LMAD x management practices x year. SMN, PTN, and yield measured from 2017 to 2019 were affected by a two-way interaction effect of rate of LMAD x management practices. The PTN, yield and ANR (N) were profoundly affected by a two-way interaction of rate of LMAD x year. ANR (N) also showed the significant main effect of management practices. SNR and AE (N) had significant main effects on the rate of LMAD, management practices, and year.

Table 2.29 ANOVA P-values for the main and interaction effects of management practice (MP), amendment treatment (LMAD), and sampling period (Year) on changes in total soil mineral nitrogen (SMN) (mg kg^{-1} soil), soil nitrogen ratio (SNR), area under curve (AUC) (mg SMN days kg $soil^{-1}$), plant total N (PTN) (kg N ha^{-1}), corn yield (kg DM ha^{-1}), agronomic efficiency (AE) (kg DM kg^{-1} N), and apparent nutrient recovery efficiency (ANR) from 2017 to 2019

	SMN	SNR	AUC	PTN	YIELD	AE (N)	ANR(N)
LMAD	<.0001	**<.0001**	<.0001	<.0001	<.0001	**0.0211**	<.0001
MP	0.0025	**0.0212**	0.0003	<.0001	<.0001	**<.0001**	**<.0001**
LMAD× MP	**0.0358**	0.3124	0.0434	**0.0213**	**0.0054**	0.5742	0.9515
Year	0.1296	**0.0021**	0.0002	<.0001	<.0001	**<.0001**	<.0001
LMAD× Year	0.2303	0.1034	0.2701	**0.0001**	**<.0001**	0.2793	**0.01**
MP × Year	0.5564	0.9708	0.0498	0.7163	0.4352	0.3373	0.9307
LMAD× MP × Year	0.2093	0.3927	**0.0367**	0.9976	0.8217	0.6829	0.9413

*Significant effects that needed multiple means comparison are shown in bold.

2.3.3.3.1 Soil Mineral Nitrogen Dynamics

The SMN concentrations measured in LMAD amended soils from 2017 to 2019 was profoundly affected by a two-way interaction of rate of LMAD × management practices (Table 2.29). The SMN in the incorporated urea treatments were almost double higher than other treatments, reaching 61.12 mg kg^{-1} soil. The SMN concentration measured across management practices at the LMAD and LMAD+U treatments showed no differences from each other but were significantly higher than the control. No differences in SMN concentrations were detected in the LMAD+U and urea treatments under the surface spreading application (Table 2.30).

Table 2.30 Effect of the two-way interaction of management practices (MP) and amendment treatment (LMAD, Urea, and control) on total soil mineral nitrogen (SMN) (mg kg^{-1} soil) from 2017 to 2019 (n=4)

MP	Amendment treatment	SMN (mg kg^{-1} soil)
Incorporation	Control	20.77 D
	LMAD	33.04 BC
	LMAD+U	39.28 BC
	Urea	61.12 A
Surface spreading	Control	20.77 D
	LMAD	28.67 CD
	LMAD+U	32.00 BC
	Urea	39.93 B

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

In both management practices, the peak SMN concentration of the LMAD+U treatments decreased over the three years from 97 mg kg^{-1} soil in July 2017 to 60 mg kg^{-1} soil by June 2019 in the incorporated treatments, and from 92 mg kg^{-1} soil to 29 mg kg^{-1} soil in surface spreading but was higher than urea in 2017 (Fig.2.17 (a) and (b)). However, under the surface spreading application, the peak SMN concentration of the LMAD treatments increased from 35 mg kg^{-1} soil to 61 mg kg^{-1} soil during the growing seasons (Fig.2.17 (b)). Moreover, the SMN concentration of the LMAD treatments significantly grew up after harvest in 2018.

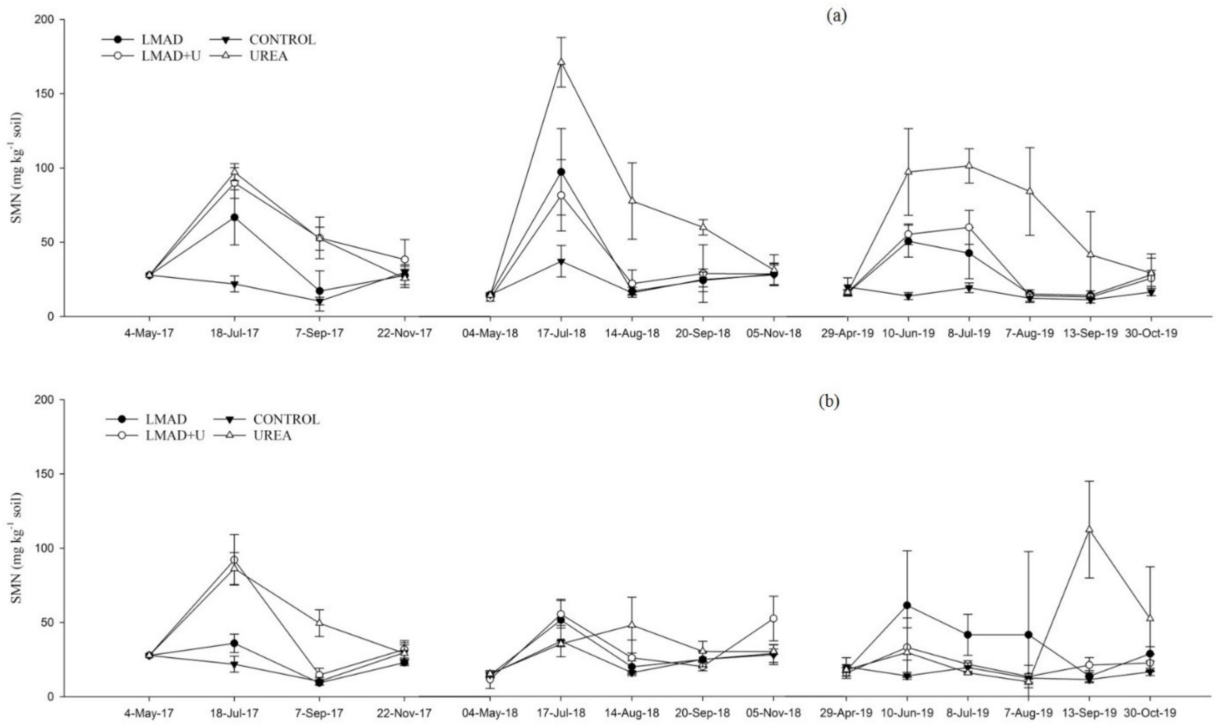


Fig. 2.17 Mean total soil mineral nitrogen (SMN) (mg kg^{-1} soil) among LMAD, LMAD+U, control and urea application across three years (a) within incorporated treatments and (b) within surface applied treatments

Area under curve values of incorporation was higher than surface spreading among urea, LMAD, and LMAD+U treatments during three growing seasons (Table 2.31). In 2017, AUC values of the LMAD+U and urea treatments were significantly higher than other treatments in both management practices. While only urea treatments in 2019 were significantly higher than other treatments in both management practices. Moreover, no differences were detected among LMAD. LMAD+U, urea, and control treatments on AUC values in the 2018 surface spreading application.

Table 2.31 Effect of the three-way interaction of management practices (MP), amendment treatment (LMAD, Urea, and control), and year on area under curve (AUC) (mg SMN days kg soil^{-1}) (n=4)

MP	Amendment treatment	Year		
		2017	2018	2019
Incorporation	Control	4190 HI	5503 FGHI	2911 I
	LMAD	7340 CDEFGH	9608 BCDE	4718 GHI
	LMAD+U	11464 B	8934 BCDEF	5806 EFGHI
	Urea	11405 B	17978 A	10169 BCD
Surface spreading	Control	4190 HI	5503 FGHI	2911 I
	LMAD	4744 GHI	6495 DEFGHI	4666 GHI
	LMAD+U	8933 BCDEF	6874 CDEFGHI	3533 HI
	Urea	10682 BC	5910 EFGHI	8522 BCDEFG

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

2.3.3.3.2 Soil nitrogen ratio

The SNR values in the incorporated application (2.3) was significantly higher than that in surface spreading (1.8). Urea treatment provided almost three times SMN concentrations in soil relative to the control, and LMAD+U and LMAD treatments were around twice greater than that of the control soil (Table 2.32).

Table 2.32 Mean soil nitrogen ratio (SNR) conducted from amendment treatment (LMAD, LMAD+U, and urea) from 2017 to 2019 (n=4)

Amendment treatment	SNR
LMAD	1.55 B
LMAD+U	1.80 B
Urea	2.82 A

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

In both management practices, the SNR values in 2017 and 2018 returned to background soil level by the end of the growing season, but the LMAD and LMAD+U treatments in 2019 were around twice greater than those of the control at the start of the growing season (Fig. 2.18 (a) and (b)). In the incorporation method, urea treatments provided the highest SMN over the three years, followed by the LMAD+U and LMAD treatments. A similar trend of SNR in 2017 was observed for the LMAD and urea treatments, while similar trends of SNR in 2018 and 2019 were observed for the LMAD and LMAD+U treatments (Fig.2.18(a)). The SNR value of the 2019 surface applied LMAD treatments had a bimodal shape in June (4.4) and August (3.4) 2019 surface (Fig. 2.18(b)).

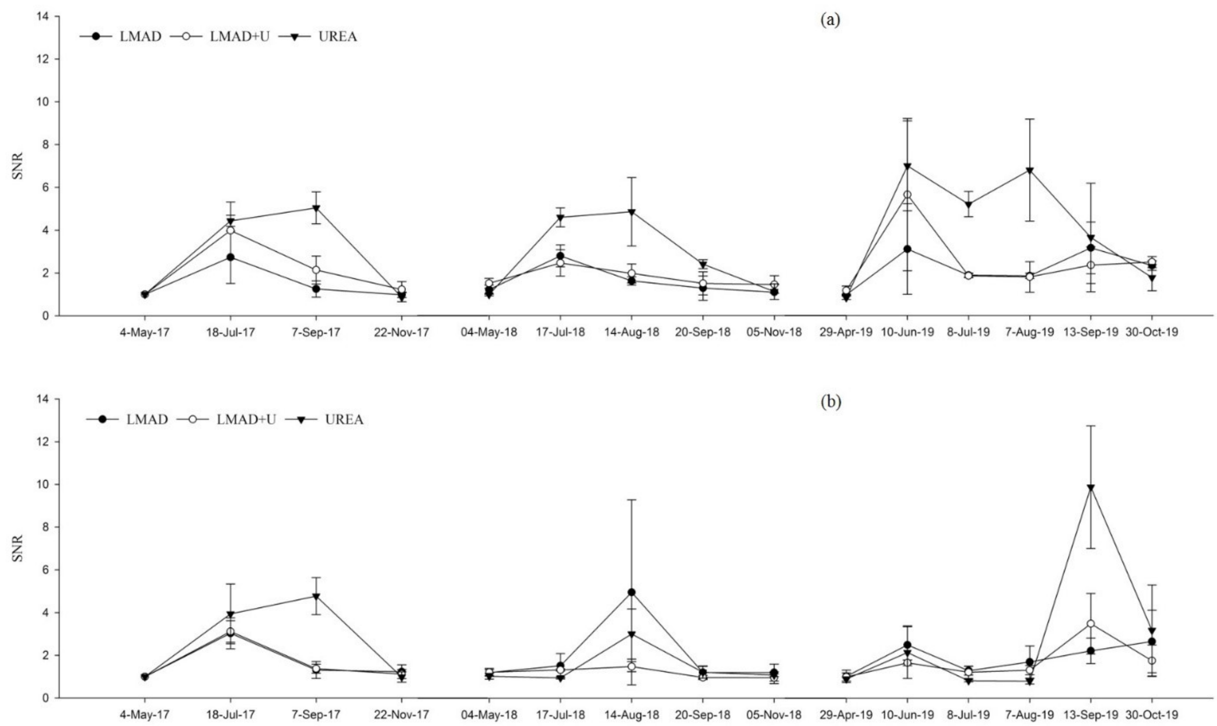


Fig. 2.18 Mean soil nitrogen ratio (SNR) in LMAD, LMAD+U, control and urea application across three years (a) within incorporated treatments and (b) within surface applied treatments

2.3.3.3.3 Crop yield

The whole-plant corn biomass yields measured under the incorporated LMAD and LMAD+U treatments were not statistically different from each other but were significantly higher than the rest of the treatments under both management practices (Table 2.33). The incorporated COMP treatments resulted in corn yields that were not significantly different from the yields of urea in the same application. Under surface spreading, biomass yield measured of the LMAD+U treatments was 14% higher than the LMAD and control treatments. There was no statistical difference between LMAD, urea, and control treatments (Table 2.33).

Table 2.33 Effect of the two-way interaction f management practices (MP) and amendment treatment (LMAD, Urea, and control) on total crop yield (kg DM ha^{-1}) from 2017 to 2019 (n=4)

MP	Amendment treatment	Yield (kg DM ha^{-1})
Incorporation	Control	11980 E
	LMAD	15817 AB
	LMAD+U	17054 A
	Urea	14848 BC
Surface spreading	Control	11980 E
	LMAD	11954 E
	LMAD+U	13642 CD
	Urea	12805 DE

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

The corn yields measured under the LMAD, LMAD+U, and urea treatments showed a decreasing trend over the three years (Table 2.34), the LMAD treatments were gradually greater than urea treatments from -3% to 4.6% over the three years, as well as LMAD+U treatments (2.5% to 16.8%). The corn yields from the LMAD+U treatments in 2017 were 51% higher than the control among years, followed by LMAD (33%) and urea (31.5%) in 2017. In 2018, no significant difference was detected in the crop yield measured among LMAD, LMAD+U, and urea treatments, but it was significantly higher than the control. In contrast, the LMAD and urea treatments resulted in the 2019 corn yield significantly lower than the control.

The highest agronomic efficiency (AE) from 2017 to 2019 was from the incorporated LMAD+U (54.12 kg DM kg^{-1} N), urea (40.85 kg DM kg^{-1} N), and LMAD+U (9.87 kg DM kg^{-1} N), respectively (Fig. 2.19). The AE of the surface application in 2019 was below 0. The AE on the LMAD+U treatment was significantly higher than that on the LMAD treatments, reached at 22.37 kg DM kg^{-1} N (Table 2.35). The AE of the incorporated application (26.43 kg DM kg^{-1} N) over the three years was significantly higher than surface application (5.01 kg DM kg^{-1} N), which were approximately 50% higher in 2017 and 539% in 2019. The AE on all treatments in both management practices showed a decreasing trend over the three years (Table 2.36). The AE showed a positive linear relationship with estimated AE under both management practices from 2017 to 2019 (R^2=0.9923) with a slop of 1.2545 (Fig. 2.20), which means 0.80 kg DM kg^{-1} N from the actual AE would be required to increase the estimated AE by 1.0 kg DM kg^{-1} N, and the estimated AE was 1.3 times higher than the actual AE.

Table 2.34 Effect of the two-way interaction of year and amendment treatment (LMAD, Urea, and control) on crop yield (kg DM ha^{-1}) over both management practices (n=4)

Amendment treatment	Year	Yield (kg DM ha^{-1})
Control	2017	13026 DEF
	2018	10539 GH
	2019	12375 EFG
LMAD	2017	17298 B
	2018	14220 CDE
	2019	10138 H
LMAD+U	2017	19693 A
	2018	15034 C
	2019	11319 FGH
Urea	2017	17128 B
	2018	14665 CD
	2019	9688 H

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

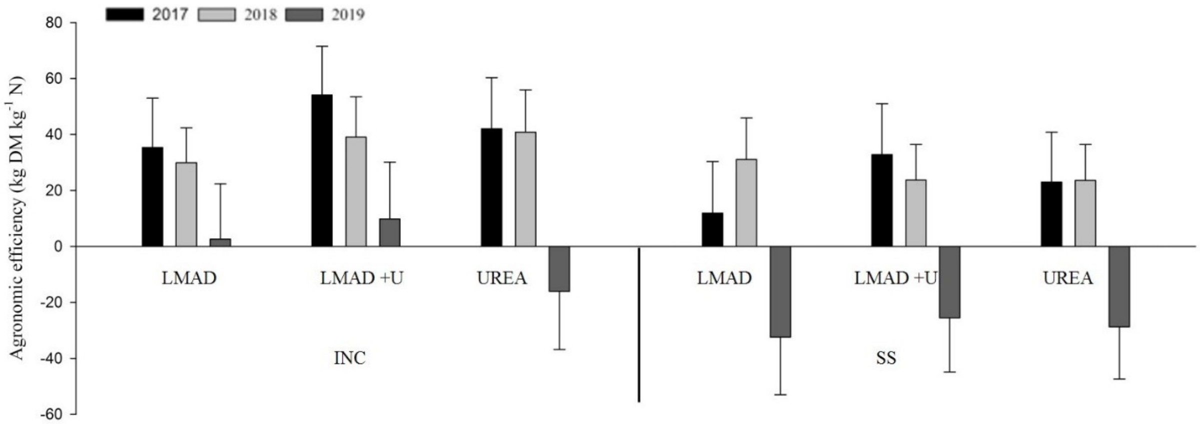


Fig. 2.19 Agronomic efficiency (kg DM kg^{-1} N) of LMAD and urea treatments in both management practices over the three years

Table 2.35 Mean agronomic efficiency (AE) (kg DM kg^{-1} N) conducted from amendment treatment (LMAD, LMAD+U, and urea) over both management practices from 2017 to 2019 (n=4)

Amendment treatment	AE (kg DM kg^{-1} N)
LMAD	10.68 B
LMAD+U	22.37 A
Urea	14.13 AB

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Table 2.36 Mean agronomic efficiency (AE) (kg DM kg^{-1} N) conducted from LMAD, LMAD+U and urea treatments over both management practices for each year (n=4)

Year	AE (kg DM kg^{-1} N)
2017	33.24 A
2018	28.99 A
2019	-15.05 B

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

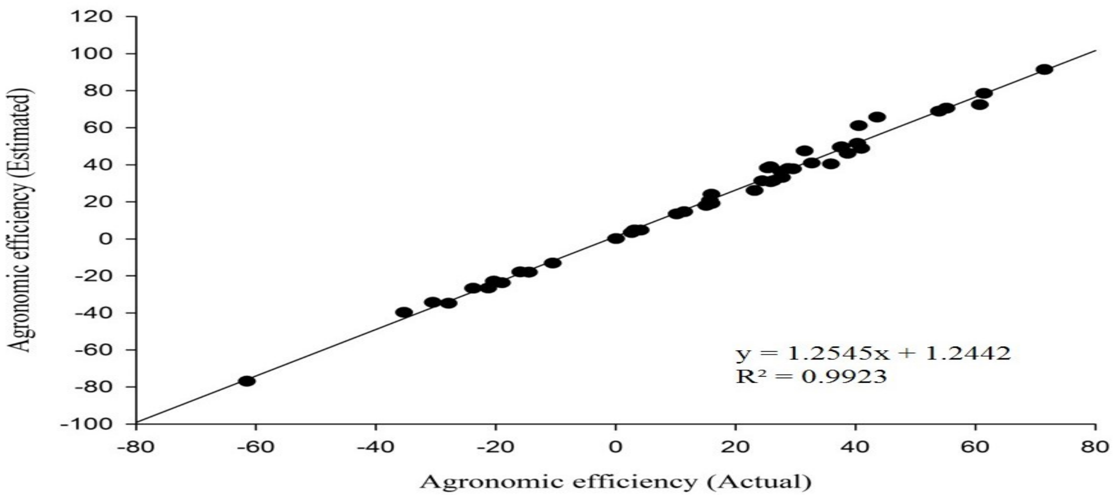


Fig. 2.20 The relationship between observed and estimated agronomic efficiency (kg DM kg^{-1} N) for the LMAD treatments under both management practices from 2017 to 2019.

2.3.3.3.4 Plant nitrogen uptake

The PTN measured over all incorporation treatments were higher than those in surface spreading (Table 2.37). Across management practices, no significant differences were detected in the incorporated LMAD, urea, and surface applied LMAD+U treatments. Under surface spreading, the PTN of the LMAD treatments was not statistically different from the control but was significantly lower than urea treatments.

The PTN measured of the LMAD+U treatments in 2017 was significantly higher than the rest of the treatments among years (Table 2.38). The PTN measured under the LMAD, LMAD+U, and urea treatments showed a decreasing trend over the three years. In 2017, there was no difference between the LMAD and urea treatments but were significantly higher than the control. The PTN measured on the 2018 LMAD treatments was not statistically different from the same treatments in 2019, as well as LMAD+U and control treatments.

Apparent nitrogen recovery (ANR) of the incorporated application (47.99%) over the three years was significantly higher than surface application (23.55%). The ANR measured on the LMAD and LMAD+U treatments had a decreasing trend over the three years (Table 2.39). The highest ANR in the LMAD amended treatments during growing seasons was the incorporated LMAD +U in 2017 at 85.68%, and the lowest was the surface applied LMAD in 2019 at -7.57% (Fig 2.21). No statistical difference was detected on ANR (N) of the LMAD treatments in the last two years, as well as LMAD+U treatments (Table 2.39). The ANR (N) showed a positive linear relationship with estimated ANR (N) under both management practices from 2017 to 2019 (R^2=0.9508) with a slope of 1.3523 (Fig. 2.22), which means 0.74% from the actual ANR(N) would be required to increase the estimated ANR(N) by 1%,and the estimated ANR(N) was 1.35 times higher than the actual ANR(N).

Table 2.37 Effect of the two-way interaction of management practices (MP) and amendment treatment (LMAD, Urea, and control) on plant total nitrogen (PTN) (kg N ha^{-1}) from 2017 to 2019 (n=4)

MP	Amendment treatment	PTN (kg N ha^{-1})
Incorporation	Control	79.34 D
	LMAD	134.21 BC
	LMAD+U	160.60 A
	Urea	136.60 B
Surface spreading	Control	79.34 D
	LMAD	92.86 D
	LMAD+U	122.12 BC
	Urea	113.98 C

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

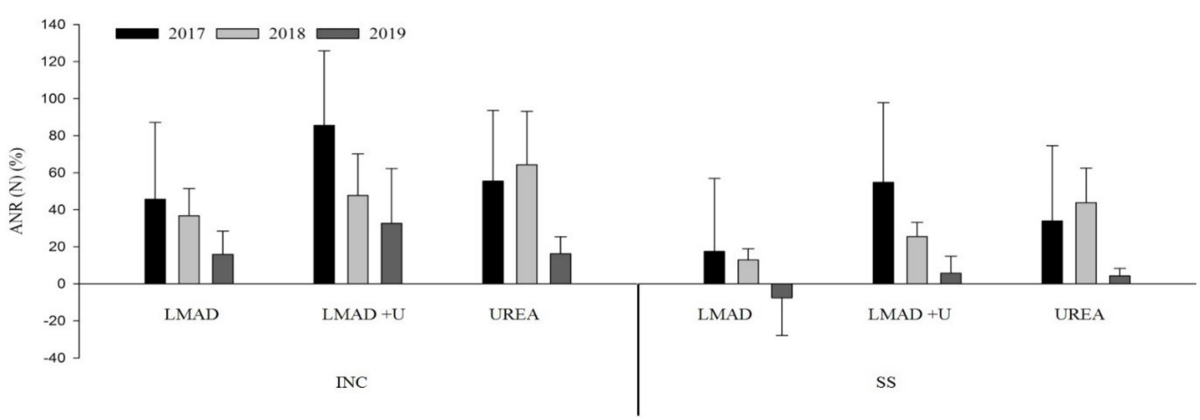


Fig. 2.21 Apparent nutrient recovery efficiency (ANR) of LMAD and urea treatments in both management practices over the three years.

Table 2.38 Effect of the two-way interaction of year and amendment treatment (LMAD, Urea, and control) on plant total nitrogen (PTN) (kg N ha^{-1}) over both management practices (n=4)

Amendment treatment	Year	PTN (kg N ha^{-1})
Control	2017	94.64 DE
	2018	59.87 F
	2019	83.52 EF
LMAD	2017	151.67 B
	2018	99.14 DE
	2019	89.80 DE
LMAD+U	2017	202.34 A
	2018	112.24 CD
	2019	109.50 CD
Urea	2017	151.00 B
	2018	129.07 BC
	2019	95.81 DE

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table 2.39 Effect of the two-way interaction of year and amendment treatment (LMAD, Urea, and control) on apparent nutrient recovery efficiency (ANR) (N) over both management practices (n=4)

Amendment treatment	Year	ANR (N)
LMAD	2017	31.61 DE
	2018	24.86 DEF
	2019	4.19 F
LMAD+U	2017	58.86 AB
	2018	36.63 CD
	2019	19.24 DEF
Urea	2017	55.12 BC
	2018	76.89 A
	2019	13.66 EF

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

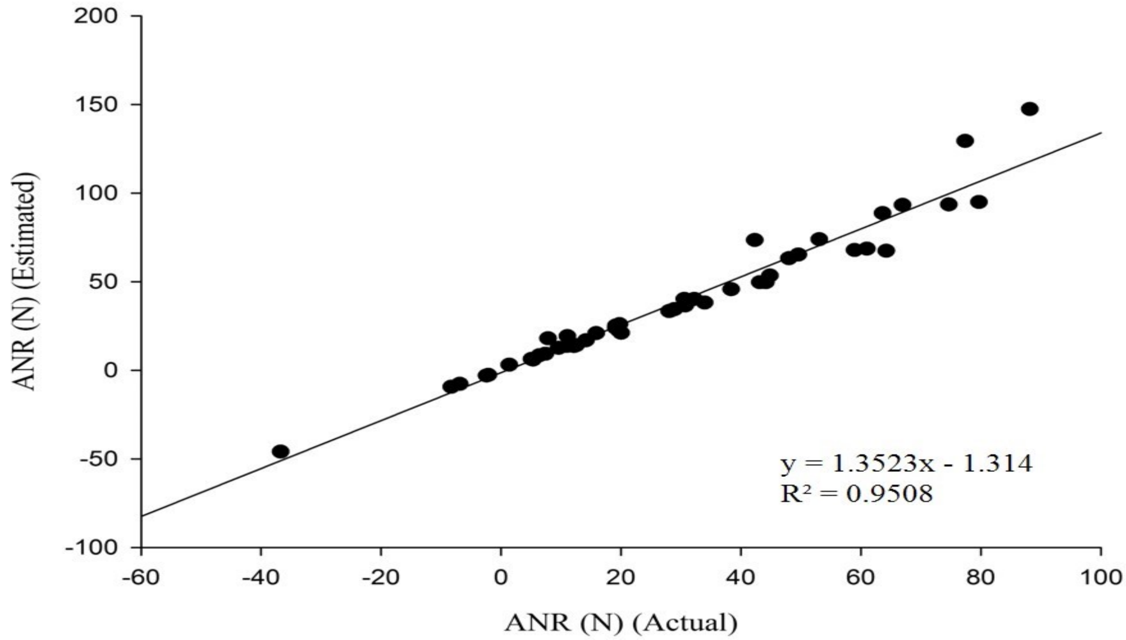


Fig. 2.22 The relationship between observed and estimated apparent nutrient recovery efficiency (N) for the LMAD treatments under both management practices from 2017 to 2019.

2.4 Discussion

In this section, the discussion was based on the comparison within each biosolid and comparing the different biosolid types. The statistical analysis for comparing the different biosolid types was in the **Appendix** (Table A1.-A12.).

2.4.1 Soil pH

The optimal pH for corn production is 6.5 or higher, but most of Nova Scotia's soils are acidic and need to be limed to bring up to 6.5 (Nova Scotia Federation of Agriculture, 2006). In this study, only the soil pH of ATB reached the optimal pH level (Fig. 2.4). The results showed that ATB treatments increased soil pH regardless of the management practices from 2017 to 2019. Christie et al. (2001) and Price et al. (2015) also determined that the annual application of ATB increased the magnitude of soil pH. In contrast, over ten years of field trials in the Pacific Northwest have determined that soil pH were similar between 0.2 to 0.4 pH unit when applying dewatered, anaerobically digested biosolids or with commercial N fertilizers (Sullivan, 2015), which means they did not significantly alter soil pH. The same results also showed on biosolids compost by Lombard et al. (2011). The mineralization in soil and plant growth is also controlled by soil pH because it directly influences microbial populations and activities; at a higher soil pH, the mineralization fraction of C and N increased, thereby increased plant uptake (Curtin et al.,1998; Neina, 2019). In our study, the total amount of SMN concentrations and crop yield in ATB amended soil were relatively higher than those in LMAD and COMP amended soils. Under lower pH conditions, N mineralization by microbes became slower, and aluminum toxicity limits root growth and phosphorus uptake and translocation to reduce the yield (Havlin et al., 2016).

2.4.2 Soil mineral nitrogen dynamics and soil nitrogen ratio

There were higher SMN concentrations measured from incorporated treatments compared to surface-applied treatments over three years. He et al. (2003) and Castillo et al. (2011) both reported that the incorporation of biosolids increased N mineralization by 60% and 25%, respectively, compared to surface application. The apparent organic mineralization from liquid digested sewage sludge of surface applied treatment and incorporated treatments were 298 and 325 g kg^{-1} (9% increase)(Adamsen & Sabey, 1987). In our study, the apparent mineralization from incorporated treatments and surface applied treatments were 40.35 mg kg^{-1} soil and 19.16 mg kg^{-1} soil, respectively. The difference in SMN concentrations may also be due to the higher N loss through the volatilization of surface applied treatments during the summer. Many studies indicated that a large amount of N from surface-applied biosolids could rapidly be lost through volatilization, and higher temperatures may also increase the loss rate (Harmel et al., 1997; Robinson & Polglase, 2000; He, 2003). Er et al. (2004) reported C:N ratio was an important factor affecting N mineralization for soil amendments and explained the 35.3% total variability of published data of N mineralization. The LMAD had the lowest C:N ratio and COMP had the highest C:N ratio in our study, which may cause the difference in SMN. There was no difference between the ATB and LMAD on SMN, while they were higher than the COMP. However, the average value of the total amount of SMN concentration measured was ATB>LMAD>COMP. Soil pH is another possible reason for the SMN effects measured in our study, particularly for ATB treatments. Some studies have indicated that raising soil pH can increase the N mineralization rate in biosolids amended soil (Hseu & Huang, 2005; Huang & Chen, 2009; Poulsen et al., 2013). Over a 48 week incubation, 3 to 34% of total N content was mineralized by biosolids and more mineral N in a pH 6.5 silt loam soil, compared to sandy soil with pH 5.7 (Hseu & Huang, 2005). Higher mineral N in ATB can also be explained by relatively poor stability due to labile organic matter, in contrast to the LMAD that already has had some organic matter digested during the anaerobic digestion process. Ives et al. (2010) reported that mineral N from lime amended biosolids increased 62% after 56 days of the soil incubation period, while only 35% of mineral N was mineralized from anaerobically digested biosolids. There was no significant difference in SMN concentrations between ATB and ATB+U treatments or between LMAD and LMAD+U treatments in both management practices. In contrast, Pawlett et al. (2015) used biosolids and urea derived organo-mineral Fertilizers (OMF) in a field-scale experiment growing ryegrass, biosolids were significantly greater than OMF on the soil total N. Averaged over three-year of samplings, SMN in the ATB+U and LMAD+U was 2% and 18% higher than that in ATB and LMAD treatments, respectively. This may have been associated with N immobilization from the larger pool of labile organic matter. Rigby et al. (2009) demonstrated total oxidized N (nitrate + nitrite) were undetectable from 0 to 48 days in a sandy silt soil with lime treated biosolids, resulting from microbial immobilization of N. Bowden et al. (2007)

calculated the mineralization rate of N from composted biosolid was -15% to -5%, which indicated a net immobilization of N. In our study, the COMP treatments showed that the smallest mineral N content compared to other biosolid types, resulting in no differences with the control soil. The biosolids produced by the composting process is a stable source of residual organic matter but with lower, more stabilized N content and availability. The COMP+U treatments resulted in higher SMN concentrations compared to the full COMP treatment as a function of the urea contribution. Han et al. (2004) concluded the interaction between composted biosolids and urea depended on the inorganic N and organic C content in the soil. The combination of urea and composted biosolids increased N mineralization, especially in a low inorganic-N soil, compared to only applying composted biosolid or urea. The AUC values, is also called as nitrogen exposure, combine the SMN concentration of multiple sampling times during the growing season. It indicates longer term effects of SMN on soil biological processes and activity of plant growth (Burton & Zebarth, 2014). In this study, the AUC values were highly related to MP and the weather conditions of each year. The AUC values over all treatments in 2017 had no differences between MP, it is possibly because urea was applied as side-dress and not incorporated into the soil. However, the incorporated treatments in 2018 were significantly higher than surface applied treatments, suggesting the effect of increased soil pH on SMN or possible cumulative effects of soil organic matter in the soil with good weather conditions or more runoff on surface applied treatments. The SMN and AUC values of all treatments in 2019 were significantly lower than the first two years due to the higher than normal precipitation in the weeks prior to planting. The high precipitation occurred in mid-May after land application of biosolid, which likely caused some nutrient leaching and runoff, particularly in the surface applied treatments. This also resulted in a later planting of the corn crop and a reduction in yields.

The SNR values provide additional information for SMN to identify the treatment effect (biosolid types and urea) compared to control. Some factors are causing the SNR values larger or lower than 1, the change of SMN pool, microorganism activities related to N mineralization and nitrification, and the loss of inorganic N (Price et al., 2015). In this study, the SNR of biosolids and urea treatments were significantly higher than the control in both management practices, suggesting biosolids and urea can increase the SMN concentrations relative to the control. Urea had the highest SNR due to the inorganic N form, which can rapidly be used by soil and corn. The urea for incorporated treatments was applied simultaneously with biosolids (Early-May) and tilled within two or three days of application to keep volatilization losses to a minimum. However, urea for surface applied treatments was side dressed after the three to four leaves growth of corn (July or August), which may cause N loss to the atmosphere due to the high temperature. The Binder et al. (2002) indicated that small amounts of SMN remained in the soil at post-harvest when 62 Mg ha^{-1}(441 kg organic N ha^{-1}) of anaerobically digested biosolids were applied to produce the maximum irrigated corn yield (28% increased over control). Similar results were found in the first two years of our study. The SNR was around 0.6 to 1.1 in the treatment plots. However, a higher SNR (1.1 to 3.2) was found in 2019, especially for urea treatments, suggesting a desynchronization between crop demands and N mineralization. Urea applications were added late in 2019, which is a possible reason that nitrogen was not delivered to the corn on time, leading to a larger post-harvest SMN surplus in the soil.

2.4.3 Crop yield and plant nitrogen uptake

The crop yield data showed that the incorporated treatments resulted in 23% higher yields than those of surface applied treatments, as well as greater N uptake (32%). Similar results have been reported by Castillo et al. (2011). The study found a 33% increase in elephant grass dry matter yield and a 31% increase in N removal for incorporated municipal biosolids compared to surface applied. However, in our study, crop yields in all treatments declined each year except for the control, indicating that other factors unrelated to biosolid types and management practices may have been involved. A similar trend was observed for N uptake of some treatments (COMP+U, LMAD, LMAD+U, and urea). Different weather conditions from year to year are influenced by corn production and nitrogen availability throughout our study. Huang et al. (2015) demonstrated that larger precipitation during the growing season negatively affected corn yield. Corn heat unit (CHU) accumulation, which is calculated by the conversion of daily maximum, and minimum air temperature, will affect dry matter yield based on different hybrid types (Kwabiah et al., 2003). In this study, the average temperature during the growing season was around 15 to 16°C. However, the CHU accumulation decreased from 2777, 2532, and 2387, and total precipitation increased from 433.6 mm, 606.7mm, and 622.4 mm over three years. Gentry et al. (2013) concluded that continuous corn production caused yield loss, and the primary factors were lower N availability and corn residue accumulation. There is no difference in SMN between 2017 and 2018 in our study, so corn residue accumulation may have contributed to decreasing the yields. The negative impacts on nutrient cycling under continuous corn production over time were found when a large amount of high C:N ratio residues was accumulated and possibly immobilized N (Green & Blackmer, 1995; Nicolardot et al., 2001). Even though increased immobilization might reduce the losses of N, but also reduced crop yield (Vanlauwe et al., 2002). Another study also reported that corn residue had the lowest N mineralization rate compared to 46 other crop residues, which resulted in a slow decomposition rate and potential for N immobilization (Trinsoutrot et al., 2000). The lower amount of crop yield in 2019 compared to the first two years was due to lower available SMN and damage to plots from hurricane Dorian, particularly in ATB, COMP, and control treatments. Crop yields of those treatments were underestimated. Therefore, the same N% in the whole plant biomass of those treatments can result in higher N uptake, which explained the N uptake in those treatments increased in 2019. The average value of total crop yields, as well as plant N uptakes, over the three years, followed the order of ATB>LMAD>COMP. These show that SMN concentrations increased when soil pH rose, thereby increasing the crop yield and N uptake. In both management practices, no differences in crop yield and N uptake were observed between ATB and ATB+U treatments, suggesting the possibility of ATB to replace the commercial fertilizer. While COMP+U was significantly higher than COMP treatments on crop yield (13%) and N uptake (27%), suggesting adding some commercial fertilizer as a supplement with biosolids compost would have a positive production effect. No difference in crop yield and N uptake was observed between the incorporated LMAD and LMAD+U treatments, while significant differences were measured in surfaced applied LMAD and LMAD+U treatments. These may be explained by nitrogen leaching easily on surface soil and poor stability of LMAD. The soil N mineralization can be roughly estimated based on the result of the SMN and plant N uptake, and the N mineralization rate was around 50% of total N for ATB, 18% for COMP, and 47% for LMAD. However, the other loss pathways of inorganic N did not account for, including volatilization, immobilization, and leaching.

2.4.4 Nutrient use efficiency

Agronomic efficiency and apparent nitrogen recovery efficiency were associated with crop yield and plant N uptake, respectively, which can provide more information to determine the efficiency of current management practices regarding the amount and timing of biosolids application as fertilizers (Zemenchik & Albrecht, 2002). The slope of the estimated AE vs. actual

AE of ATB, LMAD, and COMP were 1.5059, 1.2609, and 1.2545, respectively (Fig. 2.8, 2.14, and 2.20), which means that the estimated AE was higher than the actual AE, therefore indicated that the assumption of available N was overestimated. The AE of incorporated treatments was six times higher than that of surface applied treatments. Baligar et al. (2001) indicated that different tillage practices could increase NUE across different agro-ecosystems. Appropriate agronomic management practices can significantly improve AE, including corn planting density, timely planting, and weeding (Tittonell et al., 2007). The mean AE of corn in various regions was 24 kg kg^{-1} N, and in America was 20 kg kg^{-1} N (Cassman et al., 2002; Ladha et al., 2005). The mean of AE in incorporated treatments in 2017 (36 kg kg^{-1} N) and 2018 (30 kg kg^{-1} N) were greater than those values, while only LMAD+U and urea in surface spreading was higher, considering the better N utilization for those treatments. The low or negative AE in 2019 was due to the high crop yield of the control treatment and lower yields due to late planting and other weather-related issues over the season.

The ANR(N) can be used to estimate the relative plant N availability of the total N contained in each treatment. The ANR(N) of incorporated treatments was two times higher than that of surface applied treatments. The initial assumptions for our study were based on estimates that N availability was 50% of total N for ATB and COMP, 75% for LMAD, although even with additional urea, the ANR(N) of most treatments was average less than 40% and decreased over time. The slope of the estimated ANR vs. actual ANR of ATB, LMAD, and COMP were 1.2883, 1.489 and 1.3523, respectively (Fig. 2.10, 2.16, and 2.22), which means that the actual N availability was lower than the estimation, therefore indicated that the assumption of available N was overestimated. Ladha et al. (2005) summarized the average ANR(N) of corn was 65% in the worldwide research trials, which considers the N availability in our study should be less than assumption or have a lower level of management quality. Lower ANR(N) was due to the N losses, and the major contributions can be denitrification, ammonia volatilization, and leaching (Schlesinger, 1997; Mosier et al., 2002).

2.5 Conclusion

Applying municipal biosolids is a sustainable agricultural pathway to improve soil properties and maintain corn yield. Incorporation application can increase the SMN, crop yield, and N uptake compared to surface application. Mineral N dynamics highly differed among three types of biosolids based on their characteristics, but the SMN was greatest for ATB>LMAD>COMP. The AUC values (as nitrogen exposure) provided more information to indicate mineral N dynamics based on temporal and spatial aspects, and the SNR values showed an exact comparison between treatments and control during the growing season. Management practices also resulted in higher AUC and SNR values under incorporated treatments. Crop yield and N uptake were highly related to N availability and weather conditions, causing the reduced yield and relatively low N uptake in some treatments. Other factors, including the time of sampling and losses due to leaching or runoff, may also have played an important role in the variability of measurements in this study.

Alkaline treated biosolids in both management practices resulted in an increased soil pH during the study period. No differences were observed between ATB and ATB+U for all parameters, which indicates ATB is a potential soil amendment to substitute the commercial fertilizer. Applying COMP as slow-release fertilizer and soil conditioner resulted in better crop yields when supplemented with commercial fertilizer. Overall, the AE and ANR were greatest in the incorporated treatments, and nutrient use efficiency indicated the initial assumptions on N availability from mineralization of the three types of biosolids was overestimated.

3. Seasonal Soil Dynamics and Plant Uptake of Phosphorus and Potassium in Corn Fields Amended with Biosolids

3.1 Introduction

Biosolids are solid wastes that are treated from municipal wastewater facilities. Land application of biosolids is one beneficial option for the disposal of biosolids (CCME, 2012). The nutrient content in biosolids depends on the composition of the untreated sewage sludge, the chemicals used for purification, and the type of treatment process. In addition to nitrogen, phosphorus (P) and potassium (K) are also essential macronutrients for plant growth. The total P concentration in biosolids from different treatment processes can vary from 1 to 150 g P kg^{-1} on a dry basis (Hansen, & Chaney, 1984; Ludibeth et al., 2012; Torri et al., 2017). The total P content in alkaline treated biosolids (ATB) (3.7 to 72.6 g P kg^{-1}) and biosolids compost (COMP) (5 to 24.2 g P kg^{-1}) average on the low range of the concentrations (Corrêa, 2004; Zinati, 2004; Cooper, 2005; Barbarick & Ippolito, 2007). The total P content in anaerobically digested biosolids (AD) has been measured at > 20 g P kg^{-1} (Withers et al., 2001; Smith et al., 2006; Montgomery, 2012). The total K concentration in biosolids is from 1 to 65 g K kg^{-1} on a dry basis, which is limited (0.1 to 0.9%) due to the filtration of the soluble fraction or effluent in wastewater treatment plants (Lu et al., 2012; Bøen et al., 2013; Havlin et al., 2016). Most potassium salts are soluble, and filtration separates solid particles and wastewater (Arienzo et al., 2009). Therefore, fewer potassium compounds in the biosolids.

Large quantities of these macronutrients in biosolids are often applied to soil when rates are based on the crop N needs. For instance, a biosolid such as ATB with a P content of 1.5% and N content of 1%, added to meet an agronomic crop N requirement of 120 kg N ha^{-1}, would require

20T ha^{-1} (w/w with TS% of 60%), which would result in a P addition of 180 kg P ha^{-1}. When applied to P-deficient soils, phosphorus provided by biosolids can increase soil fertility over the long term (Zerzghi et al., 2010; Cogger et al., 2013). However, when P is already high, increasing soil P may increase the possibility of P accumulation and subsequently being lost from the field to surface water (Sullivan, 2015). Compared to other macronutrients, potassium concentrations in organic amendments are typically lower, and oversupply from biosolids to the soil does not appear to be a significant issue. Based on the various properties of different types of biosolids, it is essential to explore the response of P and K in the soil and plants, thereby to optimize and balance each element.

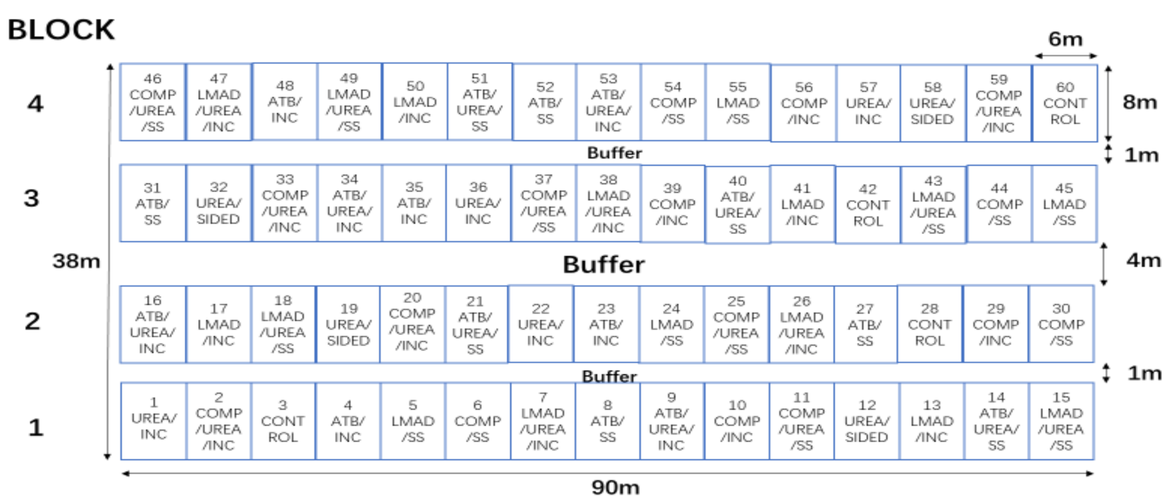
The objective of this study was to examine the influence of three differently processed biosolids, with rates applied on a nitrogen-basis and under two management practices (incorporation and surface spreading) over the three years, on seasonal soil phosphorus and potassium concentrations and annual corn plant uptake.

3.2 Materials and methods

3.2.1 Site description and treatments

A three-year field experiment was conducted using three biosolids (ATB, COMP, and liquid mesophilic anaerobically digested biosolids (LMAD)) amended to plots at a research site located in Bible Hill, Nova Scotia, Canada (45.383910, -63.242706). The soil at this site has an acidic reddish-brown sandy loam texture and is classified as an Ortho-humic Podzol under the Truro Association (CanSIS, 1991).

A 90 m x 40 m section of an agricultural research field was established and divided into 60 plots with four blocks, each block consisting of 15 plots and treatments. The plots measured 6m in width x 8m in length. The field was divided into four blocks, and the treatments were randomized within each block. Treatments consisted of three types of biosolids (ATB, COMP, and LMAD), two application methods (surface spreading and incorporation), two biosolid application rates (full and a half), one commercial fertilizer (Urea), and an unamended control. Biosolid rates, with and without urea supplementation, were applied based on a recommended N requirement for corn (120 kg ha^{-1}) from the NS Department of Agriculture. Biosolids were weighed and spread manually to achieve a uniform distribution across each plot. Biosolids were incorporated to a depth of 15cm using a tractor-mounted rototiller, and surface applied treatments were left alone after spreading. A 4 m buffer zone was established in the center of the field, between blocks 2 and 3, and 1 m buffer zones were established between the other blocks (Fig.3.1). All the plots received approximately 30 kg N at planting with the corn seed, and the actual rate of amendments/fertilizers added each year are shown in Table 3.1. Biosolid rates were based on an initial assumption of available N mineralizing over the season for ATB (50%), LMAD (75%), and COMP (50%). All the plots received 40 kg K ha^{-1} potash (0-0-50) in 2019.



*SS: Surface spreading, INC: Incorporated

Fig. 3.1 The layout of the field experiment design

Table 3.1 P and K from biosolids added to plots from 2017 to 2019 based on application rates based on corn N recommendations

	ATB			MAD			COMP		
Full rate	2017	2018	2019	2017	2018	2019	2017	2018	2019
TP applied (kg ha^{-1})	211.25	165.93	160.26	142.61	123.42	90.98	248.08	190.62	116.71
TK applied (kg ha^{-1})	141.06	141.08	54.31	7.90	18.12	42.65	42.01	36.92	52.33
Half rate									
TP applied (kg ha^{-1})	151.62	112.96	87.63	117.30	91.71	52.99	170.04	125.31	65.85
TK applied (kg ha^{-1})	70.53	73.54	40.66	3.95	12.06	34.82	21.01	21.46	39.66

3.2.2 Biosolids

An alkaline treated biosolid (ATB) was obtained from the Halifax Biosolids Facility operated by N-VIRO Systems Canada Ltd., owned by the Walker Group, in Halifax, Nova Scotia, Canada. Biosolids compost was obtained from Fundy Compost Incorporated, Brookfield, Nova Scotia, which used Class B sewage solid mixed with woody residuals to generate a Class A compost. A liquid mesophilic anaerobically digested biosolid (LMAD) was obtained from a wastewater treatment plant in St. Hyacinthe, Quebec. The properties of these three biosolids are presented in Table 3.2.

Table 3.2 Selected characteristics of three types of biosolids (n=6)

Parameters	**ATB**			**LMAD**			**COMP**		
	2017	2018	2019	2017	2018	2019	2017	2018	2019
Dry Matter (%)	61.58	61.84	62.14	22.89	23.23	17.41	42.35	41.76	44.75
pH (pH Units)	9.95	9.80	8.70	7.75	8.15	8.05	7.37	7.35	7.00
Nitrogen (%)	0.97	1.13	0.73	5.62	6.43	6.17	1.36	1.17	1.13
Ammonium-N (%)	<0.01	0.07	0.10	1.30	1.34	1.33	0.02	0.13	0.13
Calcium (%)	16.47	16.52	22.91	1.93	2.81	3.76	1.99	1.41	1.20
Potassium (%)	0.79	0.85	0.11	0.39	0.65	0.80	0.24	0.20	0.16
K2O (%)	0.96	1.03	0.13	0.47	0.79	0.97	0.29	0.24	0.19
Phosphorus (%)	0.67	0.67	0.59	2.49	3.40	3.91	0.89	0.85	0.64
P2O5 (%)	1.53	1.52	1.34	5.69	7.78	8.95	2.03	1.94	1.46
Magnesium (%)	0.31	0.31	0.10	0.30	0.35	0.45	0.18	0.17	0.16
Sodium (%)	0.08	0.08	0.04	0.21	0.31	0.42	0.05	0.05	0.04
Boron (ppm)	18.78	19.40	<10	13.69	12.14	12.03	10.87	10.72	12.32
Copper (ppm)	99.41	93.93	71.90	150.24	136.66	138.49	60.99	65.95	26.70
Iron (ppm)	7659.51	8128.46	3411.44	19806.90	35212.50	28122.20	6111.72	6550.62	6223.16
Manganese (ppm)	218.46	449.02	255.84	286.91	118.39	122.10	1172.09	1093.53	1114.38
Zinc (ppm)	207.55	248.08	178.13	212.06	237.96	280.80	175.41	198.29	123.80

3.2.3 Soil and plants sampling

Baseline sampling (Table 3.3) was conducted prior to biosolid applications, which included the collection of sixteen composite soil samples from random locations around the established field. Each composite soil sample consisted of ten soil cores taken from a 0 to 15cm depth in a 3m radius from each of the 16 areas around the field. Over the growing season, a soil composite sample was taken from each plot, consisting of ten cores collected from a depth of 0 to 15cm, approximately monthly from May to November. The soil samples were air-dried and sieved to 2mm, and the air-dried soils were bagged and stored at room temperature (20°C).

Table 3.3 Properties of baseline soil (0-15cm depth) in the experimental field (n=4)

	Soil
pH (pH Units)	5.63±0.21
Carbon (%)	1.13±0.10
Nitrogen (%)	0.20±0.02
Phosphorus (mg kg^{-1})	149.7±18.83
Potassium (mg kg^{-1})	133.95±25.81
Calcium (mg kg^{-1})	1318.73±128.94
Magnesium (mg kg^{-1})	165.95±20.15
Sodium (mg kg^{-1})	698.13±12.04
Sulphate (mg kg^{-1})	46.23±5.38
Aluminum (mg kg^{-1})	1557.98±28.37
Copper (mg kg^{-1})	3.53±0.62
Iron (mg kg^{-1})	188.55±16.83
Manganese (mg kg^{-1})	74.7±9.51
Zinc (mg kg^{-1})	1.78±0.28

*Values are presented as means ± SD (n=4)

Whole plants were harvested in the middle two rows per plot were harvested at the end of the growing season using a small plot corn harvester (The International 484, Louisville, KY, USA). Subsamples (silage) of the plant material were collected for each plot, approximately 250g, and oven-dried at 70 °C, ground using a Wiley mill, and stored at 20°C until ready for analysis.

3.2.4 Laboratory analysis

A Mehlich 3 (M3) extraction was used to determine inorganic P and K based on the current soil test methods recommended in Nova Scotia (Sims et al., 2002; Sharpley et al., 2007). Extractable P and K were determined from the soil extracts using ICP-OES at the Nova Scotia Department of Agriculture Analytical Laboratory (Bible Hill, Nova Scotia). The values are expressed as mg P kg^{-1} soil and mg K kg^{-1} soil and used to explore the changes in Mehlich-3 extractable P (M3P) and K (M3K) in soil over time.

A nitric acid microwave digestion method was used to examine total P (TP) digestion of plant samples using MARS express microwave digestor (CEM, Matthews, NC, USA) and analyzed by ICP-OES. Plant digest P and K were analyzed by ICP-OES at the Nova Scotia Department of Agriculture Analytical Laboratory, Truro, Nova Scotia, Canada (Sharpley et al., 2007). In order to examine the total plant P uptake from the soil, total plant P can be expressed as a ratio of the soil available P in order to use as an index of the relative efficiency of P utilization.

3.2.5 Nutrient use efficiency (NUE)

Apparent nutrient recovery efficiency (ANR) was used to indicate the ability of corn to absorb the applied nutrient from soil (Baligar et al., 2001):

$$ANR = \frac{Plant\ total\ nutrient\ T - Plant\ total\ nutrient\ C}{Actual\ amount\ of\ nutrient\ applied} \times 100\% \quad (1)$$

Where Plant total nutrient T refers to corn nutrient uptake from each of the 14 treatments, and Plant total nutrient C refers to nutrient uptake from the control, and the actual amount of nutrient applied refers to total nutrient applied from each of the treatments each year (Table 3.1). The unit of Plant total nutrient T and C are corn nutrient uptake (kg N ha^{-1}) of 14 treatments and the control, and the actual amount of nutrient applied from the treatments are also based on kg ha^{-1}.

3.2.6 Statistical analysis

Soil M3P, M3K, plant TP, and TK were analyzed using the PROC MIXED procedure (repeated measures) in SAS (Statistical Analysis System version 9.4, SAS Institute, Raleigh, North Carolina). Analysis of variance was carried out within biosolid types. For each biosolid type, there were two factors of interest: amendment rate, management practice, which were examined over three years of study and are considered to be fixed effects, while the block is considered to be a random effect. Significance was based on an alpha value of 0.05. Multiple means comparison, where necessary, was conducted using the Fisher Least Significant Difference test at an alpha value of 0.05.

3.3 Results

3.3.1 The effects in ATB-amended soil

The ANOVA showed a significant two-way interaction effect of management practice (MP) x year for apparent nutrient recovery efficiency (ANR K) (Table 3.4). The apparent nutrient recovery efficiency (ANR P), plant total P (PTP), Mehlich 3 Extractable elements (M3K), plant total K (PTK), and ANR (K) measured over the three years had significant interaction effects of the rate of ATB x year. PTP and ANR (P) also displayed a significant interaction effect of the rate of ATB x MP. M3P, AUC(P), and AUC(K) had significant main effects on the rate of ATB and year. PTK showed significant main effects of MP.

Table 3.4 ANOVA P-values for the main and interaction effects of management practice (MP), amendment treatment (ATB), and sampling period (Year) on changes in Mehlich 3 Extractable elements (P, and K) (mg kg^{-1} soil), area under curve (M3P and M3K) (mg days kg $soil^{-1}$) plant total P (PTP) and K (PTK) (kg ha^{-1}), and apparent nutrient recovery efficiency (P, and K) from 2017 to 2019

	M3P	AUC (P)	PTP	ANR (P)	M3K	AUC (K)	PTK	ANR (K)
ATB	**0.0018**	**0.0436**	<.0001	<.0001	<.0001	**<.0001**	<.0001	0.8956
MP	0.5771	0.6511	0.0026	0.0016	0.454	0.6669	**<.0001**	0.0001
ATB × MP	0.7705	0.8257	**0.0151**	**0.0323**	0.8802	0.937	0.062	0.272
Year	**<.0001**	**<.0001**	<.0001	<.0001	<.0001	**<.0001**	<.0001	<.0001
ATB × Year	0.4734	0.8472	**0.0017**	**0.0095**	**0.0014**	0.1097	**<.0001**	**0.0011**
MP × Year	0.751	0.8544	0.8273	0.4902	0.951	0.99	0.9196	**0.0113**
ATB × MP × Year	0.9776	0.9711	0.9025	0.4592	0.9396	0.9668	0.8645	0.9585

*Significant effects that needed multiple means comparison are shown in bold.

3.3.1.1 Mehlich 3 Extractable P and K

There were no statistical differences in the M3P concentrations measured between ATB and ATB+U treatments or the control, but these were significantly higher than the urea treatment (Table 3.5). The concentrations of M3P increased significantly across three years (Table 3.6), as well as the AUC (P) values (Table 3.8). The AUC (P) values of M3P concentration measured showed no statistical differences between the ATB and ATB+U treatments or the control, but they were also significantly higher than the urea treatment (Table 3.7).

Table 3.5 Mean M3P (mg kg^{-1} soil) conducted from amendment treatment (ATB, ATB+U, urea, and control) over both management practices from 2017 to 2019 (n=4)

Amendment treatment	M3P (mg kg^{-1} soil)
Control	202.67 AB
ATB	207.76 A
ATB+U	207.47 A
Urea	195.78 B

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Table 3.6 Mean M3P (mg kg^{-1} soil) conducted from ATB, ATB+U, urea and control treatments over both management practices in three years (n=4)

Year	M3P (mg kg^{-1} soil)
2017	169.27 C
2018	192.43 B
2019	248.55 A

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table 3.7 Mean area under curve (AUC) (mg days kg soil^{-1}) conducted from amendment treatment (ATB, ATB+U, and urea) over both management practices from 2017 to 2019 (n=4)

Amendment treatment	AUC (P)	AUC (K)
Control	37751 AB	29564 B
ATB	38995 A	38265 A
ATB+U	38941 A	37428 A
Urea	36345 B	26464 B

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table 3.8 Mean area under curve (AUC) (mg days kg soil^{-1}) conducted from ATB, ATB+U and urea treatments over both management practices in three years (n=4)

Year	AUC (P)	AUC (K)
2017	34211 C	37383 A
2018	37352 B	28917 C
2019	42461 A	32491 B

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

The M3K concentrations measured in the ATB and ATB+U treatments were not significantly different from each other over the three years, and the ATB+U treatments resulted in slightly lower M3K concentrations than the ATB treatment each year (Table 3.9). The M3K concentration measured on urea treatment in 2017 was significantly higher than that in the last two years. Moreover, the AUC values of M3K concentration (Table 3.8) decreased in 2018 and slightly increased in 2019. No significant difference in the AUC (K) between the ATB and ATB+U treatments but were significantly higher than urea and control treatments (Table 3.7).

Table 3.9 Effect of the two-way interaction of year and amendment treatment (ATB, Urea, and control) on M3K (mg kg^{-1} soil) over both management practices (n=4)

Amendment treatment	Year	M3K (mg kg^{-1} soil)
Control	2017	163.50 E
	2018	140.78 F
	2019	170.96 DE
ATB	2017	194.69 BC
	2018	184.48 CD
	2019	215.93 A
ATB+U	2017	187.05 CD
	2018	183.26 CDE
	2019	210.72 AB
Urea	2017	168.34 DE
	2018	124.21 F
	2019	134.49 F

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

3.3.1.2 Plant P uptake and K uptake

Plant P uptake in the ATB and ATB+U treatments were not significantly different from each other in either management practices or across years, but they were higher than the control (Table 3.10). The PTP measured on the incorporated ATB+U treatments resulted in a significant difference compared to the same treatment of surface spreading. The PTP measured across ATB, ATB+U, and urea treatments showed a declining trend over the three years, decreasing around 12 kg P ha^{-1} in 2018 and 5 kg P ha^{-1} in 2019 (Table 3.11). The PTP measured on the ATB treatments in 2018 and 2019 were not different from each other but lower than that in 2017, as well as in the ATB+U treatments. The urea treatments were 28% higher than the control in 2017 but were 35% lower in 2019. In both MP, the ATB+U treatments were significantly 22% and 9% higher than the ATB treatments, respectively (Table 3.12). The ATB+U treatment had a higher ANR(P) in the first two years of the study, but no difference in 2019 (Table 3.13).

Table 3.10 Effect of the two-way interaction of management practice (MP) and amendment treatment (ATB, Urea and control) on total plant total phosphorus (PTP) (kg P ha^{-1}) from 2017 to 2019 (n=4)

MP	Amendment treatment	PTP (kg P ha^{-1})
Incorporation	Control	26.02 E
	ATB	35.68 AB
	ATB+U	40.0 A
	Urea	26.78 DE
Surface spreading	Control	26.02 E
	ATB	31.45 BC
	ATB+U	30.64 CD
	Urea	26.31 DE

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Table 3.11 Effect of the two-way interaction of year and amendment treatment (ATB, Urea, and control) on PTP (kg P ha^{-1}) and PTK (kg K ha^{-1}) over both management practices (n=4)

Amendment treatment	Year	PTP (kg P ha^{-1})	PTK (kg K ha^{-1})
Control	2017	29.68 CD	121.77 CDE
	2018	21.67 EF	100.77 FG
	2019	26.70 CDE	137.86 BCD
ATB	2017	40.92 AB	177.13 A
	2018	30.83 C	136.88 BCD
	2019	28.95 CD	124.09 CDE
ATB+U	2017	45.92 A	179.85 A
	2018	31.48 C	140.41 BC
	2019	28.70 CD	117.79 DEF
Urea	2017	37.86 B	145.33 B
	2018	24.42 DE	115.26 EF
	2019	17.36 F	83.41 G

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Table 3.12 Effect of the two-way interaction of management practice (MP) and amendment treatment (ATB and ATB+U) on apparent nutrient recovery efficiency (ANR) (P) from 2017 to 2019 (n=4)

MP	Amendment treatment	ANR (P)
Incorporation	ATB	5.04 BC
	ATB+U	26.92 A
Surface spreading	ATB	1.63 C
	ATB+U	10.79 B

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Table 3.13 Effect of the two-way interaction of year and amendment treatment (ATB and ATB+U) on apparent nutrient recovery efficiency (ANR) (P) and (K) over both management practices (n=4)

Amendment treatment	Year	ANR (P)	ANR (K)
ATB	2017	9.43 B	39.25 A
	2018	1.09 B	41.65 A
	2019	-0.50 B	-50.40 B
ATB+U	2017	27.23 A	82.35 A
	2018	26.70 A	88.53 A
	2019	2.64 B	-146.89 C

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

The overall management PTK measured for incorporation (140.69 kg K ha^{-1}) was significantly higher than surface spreading (122.73 kg K ha^{-1}). The PTK measured across ATB, ATB+U, and urea treatments had a decreasing trend over the three years, decreasing around 30 kg K ha^{-1} in 2018 and 20 kg K ha^{-1} in 2019 (Table 3.11). The ATB and ATB+U treatments were not significantly different from each other for PTK from 2017 to 2019, but they were both higher than the urea and control treatments. The highest PTK measured in 2017 and 2018 was in the ATB+U and ATB, followed by the urea and control treatments. However, The PTK measured in the control treatment in 2019 was highest, followed by the ATB, ATB+U, and urea treatments. The urea treatments were 19% higher than the control in 2017 but were 39.5% lower in 2019 (Table 3.11). The ANR (K) in ATB treatments was significantly different from the ATB+U treatments in 2019 (Table 3.13). The ANR (K) for incorporation significantly different from surface spreading in 2019 but no difference in the first two years (Table 3.14).

Table 3.14 Effect of the two-way interaction of management practice (MP) and year on apparent nutrient recovery efficiency (ANR) (K)in ATB amended soil from 2017 to 2019 (n=4)

Amendment treatment	Year	ANR (K)
Incorporation	2017	76.37 A
	2018	82.45 A
	2019	-26.59 B
Surface spreading	2017	45.24 A
	2018	47.74 A
	2019	-170.70 C

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

3.3.2 The effects in COMP-amended soil

The ANOVA analysis (Table 3.15) indicates that M3P, AUC (P), PTP, ANR (P), M3K, PTK, and ANR (K) measured over the three years all had significant two-way interaction of COMP x year. The PTK and ANR (K) also displayed a significant main effect of MP. The AUC (K) values showed the significant main effect of the year.

Table 3.15 ANOVA P-values for the main and interaction effects of management practice (MP), amendment treatment (COMP), and sampling period (Year) on changes in Mehlich 3 Extractable elements (P, and K) (mg kg^{-1} soil), area under curve (M3P and M3K) (mg days kg $soil^{-1}$) plant total P (PTP) and K (PTK) (kg ha^{-1}), and apparent nutrient recovery efficiency (P, and K) from 2017 to 2019

	M3P	AUC (P)	PTP	ANR (P)	M3K	AUC (K)	PTK	ANR (K)
COMP	<.0001	<.0001	0.0926	0.4521	0.0025	0.0677	0.4035	0.4401
MP	0.3284	0.3084	0.0582	0.0707	0.3775	0.6514	**0.002**	**0.0103**
COMP × MP	0.5371	0.4912	0.4035	0.6594	0.7737	0.8919	0.274	0.187
Year	<.0001	<.0001	<.0001	<.0001	<.0001	**<.0001**	<.0001	<.0001
COMP × Year	**<.0001**	**0.0042**	**0.0002**	**0.0013**	**0.0055**	0.066	**<.0001**	**0.0001**
MP × Year	0.8802	0.8027	0.883	0.8628	0.9773	0.908	0.7871	0.8057
COMP × MP × Year	0.9853	0.9934	0.9175	0.8423	0.7864	0.8744	0.9766	0.7049

*Significant effects that needed multiple means comparison are shown in bold.

3.3.2.1 Mehlich 3 Extractable P and K

In 2017, there were no statistical differences in the M3P concentrations measured among the COMP, COMP+U, urea, and control (Table 3.16). However, the COMP and COMP+U treatment were significantly higher than urea and control treatments in the last two years. The concentrations of M3P, as well as the AUC (P) values, increased significantly across three years (Table 3.17). The AUC (P) values for M3P concentration measured showed no statistical differences between all treatments in 2017. The COMP treatments had the highest AUC (P) over the three years.

Table 3.16 Effect of the two-way interaction of year and amendment treatment (COMP, Urea, and control) on M3P (mg kg^{-1} soil) and M3K (mg kg^{-1} soil) over both management practices (n=4)

Amendment treatment	Year	M3P (mg kg^{-1} soil)	M3K (mg kg^{-1} soil)
Control	2017	170.21 I	163.50 A
	2018	188.33 FG	140.78 B
	2019	249.47 C	170.96 A
COMP	2017	176.17 HI	163.30 A
	2018	207.51 E	134.86 BC
	2019	300.18 A	171.6 A
COMP+U	2017	172.23 I	172.25 A
	2018	198.14 EF	133.45 BC
	2019	269.75 B	161.21 A
Urea	2017	166.06 I	168.34 A
	2018	184.72 GH	124.21 C
	2019	236.54 D	134.49 BC

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

The M3K concentration measured in the COMP and COMP+U treatments were not significantly different from each other across three years. However, the M3K concentrations of the COMP and COMP+U treatments in 2017 and 2019 were significantly higher than those in 2018 (Table 3.16). Moreover, the AUC values for M3K concentration (Table 3.18) decreased significantly in 2018 and slightly increased in 2019.

Table 3.17 Effect of the two-way interaction of year and Amendment treatment (COMP, Urea, and control) on area under curve (AUC) (P) (mg days kg soil^{-1}) over both management practices (n=4)

Amendment treatment	Year	AUC (P)
Control	2017	34413 FG
	2018	36617 EF
	2019	42222 C
COMP	2017	35801 FG
	2018	41476 CD
	2019	51156 A
COMP+U	2017	34897 FG
	2018	39048 DE
	2019	45795 B
Urea	2017	33553 G
	2018	35401 FG
	2019	40082 CD

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table 3.18 Mean area under curve (AUC) (K) (mg days kg soil^{-1}) conducted from COMP, COMP+U, urea, and control treatments over both management practices in three years (n=4)

Year	AUC (K)
2017	34721 A
2018	22985 C
2019	27641 B

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

3.3.2.2 Plant P uptake and K uptake

The PTP measured in both the COMP and urea treatments decreased over the study by 13 kg P ha^{-1} in 2018 and 6 kg P ha^{-1} in 2019 (Table 3.19). In contrast, the COMP+U and control treatments had slight increases from 2018 to 2019. The PTP measured among the COMP, COMP+U, and urea in 2017 were not statistically different from each other but were significantly higher than the same treatments in 2018 and 2019. The COMP and COMP+U treatments were not significantly different from each other for PTP in 2017 and 2018. There was no difference in ANR (P) for the COMP treatments over the three years but was significantly different from the COMP+U treatments in 2018 and 2019 (Table 3.20).

Table 3.19 Effect of the two-way interaction of year and amendment treatment (COMP, Urea, and control) on PTP (kg P ha^{-1}) and PTK (kg K ha^{-1}) over both management practices (n=4)

Amendment treatment	Year	PTP (kg P ha^{-1})	PTK (kg K ha^{-1})
Control	2017	29.68 B	121.77 CD
	2018	21.67 CDE	100.77 DEF
	2019	26.70 BC	137.86 ABC
COMP	2017	37.89 A	135.39 ABC
	2018	23.11 CD	100.61 DEF
	2019	26.55 BC	120.32 CDE
COMP+U	2017	39.62 A	158.73 A
	2018	26.17 BC	123.00 BCD
	2019	20.88 DE	96.97 EF
Urea	2017	37.86 A	145.33 AB
	2018	24.42 BCD	115.26 CDE
	2019	17.36 E	83.41 F

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table 3.20 Effect of the two-way interaction of management practice (MP) and year on apparent nutrient recovery efficiency (ANR) (P) and (ANR) (K) from 2017 to 2019 (n=4)

Amendment treatment	Year	ANR (P)	ANR (K)
COMP	2017	5.26 BC	32.42 BC
	2018	4.42 C	64.67 BC
	2019	-0.15 C	-69.25 C
COMP+U	2017	12.74 AB	175.94 AB
	2018	13.53 A	274.17 A
	2019	-11.45 D	-322.99 D

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Across the management practices, the PTK measured for the incorporated treatments (127.68 kg K ha^{-1}) was significantly higher than surface spreading (112.22 kg K ha^{-1}). The COMP, COMP+U, and urea treatments were not significantly different from each other in PTK in 2017 but were higher than the control treatments (Table 3.19). No differences were detected across COMP amended, urea, and control treatments in 2018. In 2019, the PTK measured from the COMP and the control treatments were significantly higher than the COMP+U and urea treatments. Across the management practices, the ANR (K) for the incorporated treatments (83%) was significantly higher than surface spreading (-31.5%). There was no difference in ANR (K) for the COMP treatments over the three years but was significantly different from the COMP+U treatments in 2018 and 2019 (Table 3.20).

3.3.3 The effects in LMAD-amended soil

Statistical analyses for M3P, AUC (P), M3K, and AUC (K) measured from 2017 to 2019 in LMAD amended soil identified by the main effects of LMAD and year (Table 3.21). The PTP, ANR (P), PTK, and ANR (K) were affected by a two-way interaction of LMAD x year, and the main effect of MP.

Table 3.21 ANOVA P-values for the main and interaction effects of management practice (MP), amendment treatment (LMAD), and sampling period (Year) on changes in Mehlich 3 Extractable elements (P, and K) (mg kg^{-1} soil), area under curve (M3P and M3K) (mg days kg $soil^{-1}$) plant total P (PTP) and K (PTK) (kg ha^{-1}), and apparent nutrient recovery efficiency (P, and K) from 2017 to 2019

	M3P	AUC (P)	PTP	ANR (P)	M3K	AUC (K)	PTK	ANR (K)
Biosolid	**<.0001**	**0.0017**	0.0012	0.0003	**0.0145**	**0.0338**	0.1518	0.0801
MP	0.741	0.6862	**0.0108**	**0.0132**	0.351	0.3437	**<.0001**	**0.0025**
Biosolid × MP	0.7954	0.747	0.0966	0.1506	0.7981	0.624	0.0766	0.2452
Year	**<.0001**	**<.0001**	<.0001	<.0001	**<.0001**	**<.0001**	<.0001	<.0001
Biosolid × Year	0.1585	0.6541	**<.0001**	**0.0105**	0.0879	0.0953	**<.0001**	**0.0004**
MP × Year	0.8934	0.9009	0.9465	0.8524	0.6107	0.9389	0.4374	0.0967
Biosolid × MP × Year	0.9999	0.9992	0.9913	0.792	0.7497	0.8994	0.8334	0.6176

*Significant effects that needed multiple means comparison are shown in bold.

3.3.3.1 Mehlich 3 Extractable P and K

The M3P concentrations measured in the LMAD treatments were not statistically different from the LMAD+U treatments, but they were 7 to 14 mg kg^{-1} soil higher than the control and urea treatments (Table 3.22). The concentrations of M3P (Table 3.23), as well as the AUC (P) values (Table 3.25), increased significantly across the three years. The AUC values based on the seasonal M3P concentrations measured showed no statistical differences between the LMAD and LMAD+U treatments, but both were 4.5% to 9% higher than the control and urea treatments (Table 3.24).

Table 3.22 Mean M3P (mg kg^{-1} soil) conducted from amendment treatment (LMAD, LMAD+U, urea, and control) over both management practices from 2017 to 2019 (n=4)

Amendment treatment	M3P (mg kg^{-1} soil)	M3K (mg kg^{-1} soil)
Control	202.67 B	158.41 A
LMAD	210.08 A	147.25 B
LMAD+U	211.48 A	142.57 B
Urea	195.78 C	142.34 B

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table 3.23 Mean M3P (mg kg^{-1} soil) conducted from LMAD, LMAD+U, urea and control treatments over both management practices in three years (n=4)

Year	M3P (mg kg^{-1} soil)	M3K (mg kg^{-1} soil)
2017	169.99 C	164.42 A
2018	192.45 B	129.41 C
2019	252.56 A	149.10 B

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table 3.24 Mean area under curve (AUC) (mg days kg $soil^{-1}$) conducted from amendment treatment (LMAD, LMAD+U, and urea) over both management practices from 2017 to 2019 (n=4)

Amendment treatment	AUC (P)	AUC (K)
Control	37751 BC	29564 A
LMAD	39454 AB	26368 B
LMAD+U	39619 A	26939 B
Urea	36345 C	26464 B

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table 3.25 Mean area under curve (AUC) (mg days kg soil^{-1}) conducted from LMAD, LMAD+U and urea treatments over both management practices in three years (n=4)

Year	AUC (P)	AUC (K)
2017	34425 C	34276 A
2018	37347 B	21877 C
2019	43105 A	25848 B

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

The M3K concentrations measured among the COMP, COMP+U, and urea treatments were not statistically different but were significantly lower than the control (Table 3.22). The highest M3K concentrations measured in three years was 164.42 mg kg^{-1} soil in 2017, followed by 2019 and 2018 (Table 3.23). Moreover, the highest AUC(K) was in the control treatment and no significant difference among the LMAD, LMAD+U, and urea treatments (Table 3.24). The AUC values for M3K concentrations indicated similar trends in LMAD amended soil over the three years, which decreased significantly in 2018 and slightly increased in 2019 (Table 3.25).

3.3.3.2 Plant P uptake and K uptake

The PTP measured from the incorporated treatments (29.05 kg P ha^{-1}) was significantly higher than surface spreading (26.38 kg P ha^{-1}). The PTP measured among the LMAD, LMAD+U, and urea treatments showed a decreasing trend over the three years (Table 3.26). There was no difference between the LMAD+U and urea treatments in PTP but were significantly higher than the control in 2017. The PTP measured on the LMAD and LMAD+U treatments were not statistically different from each other in 2018 and 2019. Applying LMAD in 2019 resulted in a 7.5% lower PTP than control. The ANR (P) for the incorporated treatments (22%) was significantly higher than surface spreading (8.5%). The ANR (P) for the LMAD+U treatments were 40.7% and 21.13% higher than the LMAD treatments in 2017 and 2018, respectively (Table 3.27).

Table 3.26 Effect of the two-way interaction of year and amendment treatment (LMAD, Urea, and control) on PTP (kg P ha^{-1}) and PTK (kg K ha^{-1}) over both management practices (n=4)

Amendment treatment	Year	PTP (kg P ha^{-1})	PTK (kg K ha^{-1})
Control	2017	29.68 C	121.77 CD
	2018	21.67 EFG	100.77 DEF
	2019	26.70 CD	137.86 BC
LMAD	2017	35.17 B	152.02 AB
	2018	25.54 CDE	109.16 DE
	2019	20.03 FG	86.50 F
LMAD+U	2017	42.72 A	166.64 A
	2018	28.14 CD	119.65 CD
	2019	23.29 DEF	96.25 EF
Urea	2017	37.86 AB	145.33 B
	2018	24.42 DEF	115.26 DE
	2019	17.36 G	83.41 F

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Across the management practices, the PTK measured for the incorporated treatments (128.56 kg K ha^{-1}) was significantly higher than surface spreading (110.54 kg K ha^{-1}). The PTK measured all treatments showed a decreasing trend over the three years except the control (Table 3.26). The LMAD and LMAD+U treatments were not significantly different from each other for PTK in the first two years but were significantly higher than other treatments. No statistical differences were detected across LMAD amended, urea, and control treatments in 2018. The PTK measured on the control treatment was significantly higher than those of other treatments in 2019. The ANR (K) in the incorporation (522%) was significantly higher than the surface spreading (131%). There was no difference in ANR (K) for the LMAD treatments over the three years but were significantly different from the LMAD+U treatments in 2017 and 2019 (Table 3.27). The ANR (K) for all treatments in 2017 and 2018 were over 100%.

Table 3.27 Effect of the two-way interaction of management practice (MP) and year on apparent nutrient recovery efficiency (ANR) (P) and (ANR) (K) from 2017 to 2019 (n=4)

Amendment treatment	Year	ANR (P)	ANR (K)
LMAD	2017	10.84 B	382.95 BC
	2018	12.94 B	235.51 BC
	2019	-8.78 C	37.61 C
LMAD+U	2017	51.56 A	1136.16 A
	2018	34.07 A	644.18 B
	2019	-8.98 C	-474.31 D

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

3.4. Discussion

In this section, the discussion was based on the comparison within each biosolid and comparing the different biosolid types. The statistical analysis for comparing the different biosolid types was in the **Appendix** (Table A13.-A20.).

3.4.1 Mehlich 3 Extractable P and K

The M3P and M3K concentrations measured in the baseline soil in 2017 before the application was 149.71 mg P kg^{-1} and 134 mg K kg^{-1}, respectively. These values are in the high range based on the Soil Test Interpretation Ratings for Nova Scotia Crops (Nova Scotia Department of Agriculture, 2011). It indicates that P and K amendments of manure and chemical fertilizer may contribute to the high M3P and M3K concentration due to the previous management history at this site.

The results for the study show that M3P concentrations for the amended soils, as well as AUC(P), had an increasing trend across the three years of applications, but no differences between management practices were observed. The possible location for the added P from biosolids treatments is in the plant biomass and grain or losses through runoff and leaching. Applying the full rate and half rate with urea supplement in three types of biosolids did not increase the M3P significantly during the three growing seasons. Soil sorption is one possible way that added P becomes unavailable, in addition to binding into other P forms. Around 50% of P in biosolids is available for plant uptake in the first-year application, but inorganic P from biosolids quickly interacts with minerals in the soil to become fixed-P, and mineralization into plant-available forms takes time (Jenkins, 2000; Douglas, 2002). Leachate P from P-deficient acid Florida sandy soils amended with N-Viro (ATB), anaerobically digested, and composted biosolids were less than 1% of applied P (Elliott et al., 2002). Withers et al. (2001) demonstrated that a lower risk of P runoff existed after applying biosolids (liquid anaerobically digested) compared to triple superphosphate and liquid cattle manure at a similar P application rate. Urea effect is also another possible reason between full rate and half rate treatments. Higher mineralization of organic P in biosolids was under the influence of urea, even adding the half supply. There was no difference in M3P in the first two among three types of biosolids. The average value of the total amount of M3P, over the three years, followed the order of COMP>LMAD>ATB>control>urea. Low P uptake of COMP may cause high M3P availability in the soil. An 84-days canola pot experiment comparing swine manure and urea on soil P indicated the available P in urea treatment was significantly lower than unfertilized soils (Qian & Schoenau, 2000). Adding N to the soil significantly change the ratio of soil N:P and accelerate of P immobilization (Barrow, 1960). However, various parameters, such as climate, topography, management practices, and drainage, may have interacted to cause some results during the study period.

The M3K concentration measured in 2019 for all treatments increased compared to that in 2018, potentially due to a late-season fertilizer addition. It was applied 40 kg K ha^{-1} potash (0-0-50) in 2019; otherwise, the M3K concentrations measured may show a decreasing trend over three years, and no differences between management practices were observed. Incorporation allowed fertilizer to more in contact with the soil, and some soil minerals formed strong complexes with K in the interlayer area, so released K slowly, which possibly cause the K fixation (Mahle, 2001; Pettygrove et al., 2011). The total amount of M3K over the three years followed the order of ATB>COMP>LMAD, but no differences between full rate and half rate with urea supplement, considering the enhancement effect of urea. Some studies indicated that alkaline materials increased soil M3K (Cabral et al., 2008; Gagnon & Ziadi, 2012). Rens et al. (2018) indicated the positive relationship between K adsorption and Ca desorption by a sandy soil with biosolid amendments. In our study, the Ca content of ATB was around 16 to 23%, which was ten times higher than COMP and six times higher than LMAD. This may explain the higher M3K in ATB compared to another two biosolids.

3.4.2 Plant P and K uptake

Plant P and K uptake results showed that all incorporated treatments were 15% and 19% higher than those of surface applied treatments, respectively. This suggests that incorporation enhances the plant to uptake more nutrients from soil, which was the same result from Chatterjee & Lal (2009). Incorporated application placed the fertilizer near the seeding root zone so nutrients can be easily uptake by plants, and also reduced P runoff (Mahle, 2001; Daverede et al., 2004). Leikam et al. (1983) indicated incorporated P (40 lb P_2O_5/ac) with 75 lb N/ac in dryland winter wheat produced twice as much grain (29 vs. 14 bu/ac) than when the same fertilizer was surface applied. A three-year, fifteen sites study on K placement effects on corn found the grain yield in incorporated treatments was ten bu/ac higher than surface spreading (Bordoli & Mallarino, 1998). A decrease occurred in PTP and PTK over the three years except for the COMP and control treatments. Schlegel & Havlin (2017) found that the P uptake for irrigated continuous corn significantly affected nitrogen supply and uptake. Reduced P uptake can be explained by the positive interaction of N x P and the reduced N availability (Antille et al., 2013; Antille et al., 2014). High M3P concentrations in the soil also increased the osmotic pressure and destroyed the root tip meristem on plants, possibly resulting in lower P uptake (Costa et al., 2009). Johnston & Milford (2007) and Hirniak (2018) indicated that a positive interaction between N and K in corn existed; enhanced K uptake depended on the increase of N availability. Therefore, the decline of PTK can be explained by the decreasing N uptake trend in our study. There was no difference between the LMAD and COMP treatments on PTP, but it was significantly lower than ATB treatments. The average value of the total P uptake was ATB>LMAD>COMP over three years. Alkaline treated biosolids increased the soil pH, which reduced the adsorption of P by reducing the activity of exchangeable Al and Fe and increased the available P of plants in the soil (Iyamuremye & Dick, 1996; Havlin et al., 2016). The total K uptake was ATB>LMAD>COMP in first two years and COMP in 2019 was higher than LMAD, but no difference was observed between full rate and half rate with urea supplement in both management practices. The highest K uptake in ATB amended soil can be explained by the higher K content in ATB.

3.4.3 Nutrient use efficiency

The management practices had a significant effect on ANR (P) and ANR (K), resulting in higher efficiency on incorporation. The ANR (P) and ANR (K) of half-rate biosolids with urea in most treatments in 2017 and 2018 were higher than full rate biosolids, and it suggests adding urea also possibly influenced P and K uptake. A pot experiment in the greenhouse was conducted with higher P and K uptake of corn in urea+ manure (50%:50%) treatments than manure alone when nutrient was based on the N requirement (200 kg ha^{-1}) (Irshad et al., 2002). Syers et al.

(2008) concluded that most of the benefits of applying P and K fertilizers to many soils occurred in the subsequent years due to the impact on soil fertility. In our study, the ANR (P) and ANR (K) of most treatments slightly increased in 2018 compared to 2017. While in 2019, either P or K recoveries were low, even negative, resulting in the low nutrient absorption by corn. It was due to the poor rainfall distribution and hurricane damage. Most crops can recover 20 to 30% of P applied under suitable growth conditions (Dobermann, 2007), but the ANR (P) of all full rate treatments over the three years were lower than 20%. Some factors relative to corn growth affect the P uptake, such as soil P availability. García (2004) found that high ANR(P) can be measured when the soil fertility is far below the critical level, and they decreased rapidly as the soil fertility increased in wheat experiments in Argentina. Moreover, the ANR (K) of some treatments was over 100% in 2017 and 2018, especially the COMP+U, LMAD, and LMAD+U treatments, which indicates that the ANR (K) in this study not due to the biosolids application and also from soil K.

3.5 Conclusion

Our results indicate that applying municipal biosolids based on N requirements in soils resulted in an increase in M3P and a decrease in M3K over three consecutive years of applications in a corn cropping system. No differences in M3P and M3K between incorporated or surface application practices were observed. This resulted in P cumulation and potential K fixation in the soil. The higher M3K content in ATB amended soil was observed compared to the other two types of biosolids. When compared across the management practices, incorporation resulted in higher P and K uptake than the surface spreading. No difference was observed between full rate and half rate with urea supplement in both management practices, suggesting urea combined with biosolids can enhance the P and K uptake by corn.

4. Conclusion

This study shows that applying municipal biosolids is a sustainable agricultural way to improve soil properties (soil pH and nutrient availability) and maintain corn yield. It also indicated a better understanding of the interaction between biosolids characteristics and nutrient availability in amended soils and compared the performance of different municipal biosolids. This can help farmers to select the suitable biosolid types for the application and also enable people to develop practical guidelines to increase crop use efficiency of applied nutrients.

The soil pH results in this study indicated that ATB was an alternative fertilizer in acid soil, but farmers should consider the ATB rate applied to the soil, which can reach the optimum level of crop growth. LMAD, COMP, and urea did not significantly alter soil pH.

The total amount of SMN measured was ATB>LMAD>COMP, which considers the increased soil pH was the primary factor. The SMN measured were greater in incorporation compared to surface spreading, suggesting larger N loss by volatilization in surface applied treatments. No difference in SMN concentration between the ATB and ATB+U treatments or between the LMAD and LMAD+U treatments in both management practices, resulting in the potential N immobilization. When applying COMP as fertilizer, farmers need to adjust the amount or add other commercial fertilizers to increase N availability.

The M3P showed an increasing trend over the three years, and no differences between management practices were observed, suggesting potential P accumulation in the soil. Soil sorption of P and P loss through leaching may cause no difference between full rate and half rate with urea supplement in three types of biosolids. No management practices and rates effect were observed on M3K as well, suggesting urea possibly enhances the K availability. The M3K concentrations measured in ATB amended soil were significantly higher than others due to the positive relationship between K adsorption and Ca desorption in soil.

Crop responses highly depend on N availability and weather conditions. Crop yield and nutrient (N, P, and K) uptake results in this study showed that incorporation was better than surface spreading. Crop yield showed a decreasing trend over the three years, caused by increased precipitation and reduced corn heat unit. The crop responses were greatest for ATB>LMAD>COMP over the three years, which can be explained by increased soil pH, thereby raising plant available nutrient and crop uptake. Reduced P and K uptake were associated with reduced N availability over time. Based on the results of nutrient use efficiency, the assumption of N availability in three biosolids was overestimated. Overall, alkaline treated biosolids have the best performance in this study.

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Appendix Supplemental Data

1. Nitrogen

Table A1. ANOVA P-values for the main and interaction effects of management practice (MP), biosolid types, and sampling period (Year) on changes in total soil mineral nitrogen (SMN) (mg kg^{-1} soil), soil nitrogen ratio (SNR), area under curve (AUC) (mg SMN days kg $soil^{-1}$), plant total N (PTN) (kg N ha^{-1}), corn yield (kg DM ha^{-1}), agronomic efficiency (AE) (kg DM kg^{-1} N), and apparent nutrient recovery efficiency (ANR) from 2017 to 2019

	SMN	SNR	AUC	PTN	YIELD	AE (N)	ANR(N)
Biosolid	<.0001	**<.0001**	<.0001	**<.0001**	<.0001	0.0249	<.0001
MP	<.0001	**0.0003**	<.0001	**0.0002**	<.0001	**<.0001**	**<.0001**
Biosolid × MP	**0.0347**	0.2807	0.0191	0.0743	**0.0027**	0.7447	0.1684
Year	**0.0211**	**<.0001**	<.0001	**<.0001**	<.0001	<.0001	<.0001
Biosolid × Year	0.8719	0.177	0.8923	0.2102	**<.0001**	**<.0001**	**0.0002**
MP × Year	0.1884	0.4418	0.0001	0.4287	0.6002	0.5248	0.7824
Biosolid × MP × Year	0.285	0.3335	**0.0096**	0.8123	0.9218	0.4506	0.8345

*Significant effects that needed multiple means comparison are shown in bold.

Table A2. Effect of the two-way interaction of management practice (MP) and biosolid types on SMN (mg kg^{-1} soil) from 2017 to 2019 (n=4)

MP	Biosolid types	SMN (mg kg^{-1} soil)
Incorporation	Control	20.77 E
	ATB	38.90 B
	COMP	29.66 CD
	LMAD	36.16 BC
	Urea	61.12 A
Surface spreading	Control	20.77 E
	ATB	32.91 BC
	COMP	24.03 DE
	LMAD	30.34 CD
	Urea	39.93 B

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table A3. Mean SMN (mg kg^{-1} soil) conducted from all treatments over both management practices for each year (n=4)

Year	SMN (mg kg^{-1} soil)
2017	35.14 A
2018	35.26 A
2019	29.98 B

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table A4. Effect of the hree-way interaction of management practice (MP), biosolid types, and year on area under curve (AUC) (mg days kg $soil^{-1}$) (n=4)

MP	Biosolid types	Year		
		2017	2018	2019
Incorporation	Control	4169 HIJK	5556 FGHIJK	3051 JK
	ATB	7967 CDEF	10802 B	5939 FGHIJ
	COMP	5755 FGHIJ	8061 CDEF	4366 IJK
	LMAD	9368 BCD	9326 BCD	5466 GHIJ
	Urea	11366 B	18069 A	10523 BC
Surface spreading	Control	4169 HIJK	5556 FGHIJK	3051 JK
	ATB	7427 DEFG	6889 EFGH	5054 GHIJK
	COMP	5871 FGHIJ	5336 GHIJK	2961 K
	LMAD	6819 EFGH	6747 EFGHI	4315 JK
	Urea	10642 BC	5970 EFGHIJ	9346 BCDE

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table A5. Mean soil nitrogen ratio (SNR) conducted from biosolid types over both management practices from 2017 to 2019 (n=4)

Biosolid types	SNR
ATB	1.82 B
COMP	1.31 C
LMAD	1.68 B
Urea	2.82 A

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table A6. Mean soil nitrogen ratio (SNR) conducted from all treatments over both management practices for each year (n=4)

Year	SNR
2017	1.91 B
2018	1.54 C
2019	2.27 A

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table A7. Effect of the two-way interaction of management practice (MP) and biosolid types on crop yield (kg DM ha^{-1}) from 2017 to 2019 (n=4)

MP	Biosolid types	Yield (kg DM ha^{-1})
Incorporation	Control	11980 E
	ATB	17421 A
	COMP	14907 B
	LMAD	16436 A
	Urea	14848 BC
Surface spreading	Control	11980 E
	ATB	13687 CD
	COMP	11773 E
	LMAD	12798 DE
	Urea	12805 DE

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table A8. Effect of the two-way interaction of year and biosolid types on crop yield (kg DM ha^{-1}) over both management practices (n=4)

Biosolid types	Year	Yield (kg DM ha^{-1})
Control	2017	13026 EF
	2018	10539 HI
	2019	12375 FGH
ATB	2017	18131 A
	2018	15214 CD
	2019	13315 EF
COMP	2017	16244 BC
	2018	12594 FG
	2019	11183 GHI
LMAD	2017	18096 A
	2018	14627 DE
	2019	10729 HI
Urea	2017	17128 AB
	2018	14665 CDE
	2019	9688 I

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table A9. Effect of the two-way interaction of year and biosolid types on agronomic efficiency (AE) (kg DM kg^{-1} N) over both management practices (n=4)

Biosolid types	Year	AE (kg DM kg^{-1} N)
ATB	2017	24.52 B
	2018	24.09 B
	2019	5.06 C
COMP	2017	25.11 B
	2018	11.58 C
	2019	-6.75 D
LMAD	2017	34.95 A
	2018	27.36 AB
	2019	-11.37 DE
Urea	2017	32.55 AB
	2018	32.23 AB
	2019	-22.40 E

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Table A10. Mean plant total N (PTN) (kg N ha^{-1}) conducted from biosolid types over both management practices from 2017 to 2019 (n=4)

Biosolid types	PTN (kg N ha^{-1})
Control	79.34 D
ATB	156.00 A
COMP	101.82 CD
LMAD	127.45 B
Urea	125.29 BC

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Table A11. Mean plant total N (PTN) (kg N ha^{-1}) conducted from all treatments over both management practices for each year (n=4)

Year	PTN (kg N ha^{-1})
2017	152.41 A
2018	98.05 B
2019	103.47 B

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Table A12. Effect of the two-way interaction of year and biosolid types on apparent nutrient recovery efficiency (ANR) (N) over both management practices (n=4)

Biosolid types	Year	ANR (N)
ATB	2017	42.79 AB
	2018	29.14 B
	2019	34.27 B
COMP	2017	16.00 C
	2018	11.15 C
	2019	5.09 C
LMAD	2017	47.48 A
	2018	30.74 B
	2019	11.71 C
Urea	2017	39.37 AB
	2018	54.07 A
	2019	10.24 C

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

2. Phosphorus and potassium

Table A13. ANOVA P-values for the main and interaction effects of management practice (MP), biosolid types, and sampling period (Year) on changes in Mehlich 3 Extractable elements (P, and K) (mg kg^{-1} soil), area under curve (M3P and M3K) (mg days kg $soil^{-1}$) plant total P (PTP) and K (PTK) (kg ha^{-1}), and apparent nutrient recovery efficiency (P, and K) from 2017 to 2019

	M3P	AUC (P)	PTP	ANR (P)	M3K	AUC (K)	PTK	ANR (K)
Biosolid	<.0001	**<.0001**	<.0001	0.0033	<.0001	<.0001	<.0001	<.0001
MP	0.2914	0.3064	**0.0005**	**0.0008**	0.1744	0.2942	**<.0001**	0.0003
Biosolid × MP	0.7621	0.7892	0.1071	0.37	0.877	0.849	0.1273	**0.0256**
Year	<.0001	**<.0001**	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Biosolid × Year	**<.0001**	0.1099	**0.0061**	**0.0117**	**<.0001**	**0.0023**	**<.0001**	**<.0001**
MP × Year	0.7371	0.7794	0.9765	0.7932	0.8707	0.9561	0.7895	0.3491
Biosolid × MP × Year	0.9994	0.9977	0.9894	0.9664	0.8986	0.9595	0.9168	0.1287

*Significant effects that needed multiple means comparison are shown in bold.

Table A14. Effect of the two-way interaction of year and biosolid types on M3P and M3K (mg kg-1 soil) over both management practices (n=4)

Biosolid types	Year	M3P (mg kg^{-1} soil)	M3K (mg kg^{-1} soil)
Control	2017	170.21 I	163.50 D
	2018	188.33 FG	140.78 EF
	2019	249.47 C	170.96 CD
ATB	2017	170.41 I	190.87 B
	2018	198.34 EF	183.87 BC
	2019	254.09 C	213.32 A
COMP	2017	174.2 HI	167.77 D
	2018	202.82 E	134.16 EF
	2019	284.97 A	166.41 D
LMAD	2017	171.85 I	162.92 D
	2018	198.38 EF	126.33 F
	2019	262.11 B	145.47 E
Urea	2017	166.06 I	168.34 CD
	2018	184.72 GH	124.21 F
	2019	236.54 D	134.49 EF

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Table A15. Mean area under curve (AUC) (P) (mg days kg $soil^{-1}$) conducted from biosolid types over both management practices from 2017 to 2019 (n=4)

Biosolid types	AUC(P)
Control	37751 CD
ATB	38968 BC
COMP	41362 A
LMAD	39537 B
Urea	36345 D

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Table A16. Mean area under curve (AUC) (P) (mg days kg $soil^{-1}$) conducted from all treatments over both management practices for each year (n=4)

Year	AUC(P)
2017	34524 C
2018	37932 B
2019	43921 A

* Different letters indicate the significant differences based on LSMeans ($p<0.05$), values sharing the same letter within the column are not significantly different.

Table A17. Effect of the two-way interaction of year and biosolid types on area under curve (AUC) (K) (mg days kg soil^{-1}) over both management practices (n=4)

Biosolid types	Year	AUC(K)
Control	2017	33499 CD
	2018	24487 FGH
	2019	30707 DE
ATB	2017	40251 A
	2018	35314 BC
	2019	37974 AB
COMP	2017	34928 BC
	2018	23451 GH
	2019	28275 EF
LMAD	2017	34038 CD
	2018	21236 H
	2019	24687 G
Urea	2017	35530 BC
	2018	20551 H
	2019	23310 GH

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table A18. Effect of the two-way interaction of year and biosolid types on PTP and PTK (kg ha^{-1}) over both management practices (n=4)

Biosolid types	Year	PTP (kg P ha^{-1})	PTK (kg K ha^{-1})
Control	2017	29.68 CD	121.77 DEFG
	2018	21.67 FG	100.77 GH
	2019	26.70 CDEF	137.86 CDE
ATB	2017	43.42 A	178.49 A
	2018	31.16 C	138.64 CD
	2019	28.82 CD	120.94 EF
COMP	2017	38.75 B	147.06 BC
	2018	24.64 DEF	111.80 FG
	2019	23.72 EF	108.64 FG
LMAD	2017	38.95 B	159.33 B
	2018	26.84 DE	114.4 FG
	2019	21.66 FG	91.38 H
Urea	2017	37.86 B	145.33 BC
	2018	24.42 DEF	115.26 EFG
	2019	17.36 G	83.41 H

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table A19. Effect of the two-way interaction of year and biosolid types on apparent nutrient recovery efficiency (ANR) (P and K) over both management practices (n=4)

Biosolid types	Year	ANR (P)	ANR (K)
ATB	2017	18.33 BC	60.80 CD
	2018	13.90 BC	65.09 CD
	2019	1.07 DE	-98.65 DE
COMP	2017	8.99 CD	104.18 CD
	2018	8.98 CD	169.42 C
	2019	-5.80 E	-196.12 E
LMAD	2017	31.20 A	759.55 A
	2018	23.50 AB	439.84 B
	2019	-8.88 E	-218.35 E

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.

Table A20. Effect of the two-way interaction of management practice (MP) and biosolid types on apparent nutrient recovery efficiency (ANR) (K) from 2017 to 2019 (n=4)

MP	Biosolid types	ANR (K)
Incorporation	ATB	44.08 B
	COMP	83.18 B
	LMAD	522.92 A
Surface spreading	ATB	-25.91 B
	COMP	-31.52 B
	LMAD	131.11 B

* Different letters indicate the significant differences based on LSMeans (p<0.05), values sharing the same letter within the column are not significantly different.