## ORIGINAL ARTICLE

# Soil nitrogen mineralization in a soil with long-term history of fresh and composted manure containing straw or wood-chip bedding

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Abstract Long-term effects of fresh (FM) versus composted (CM) beef manure application to barley (Hordeum vulgare L.) on potentially mineralizable nitrogen  $(N_0)$ , and mineralizable nitrogen  $(N)$  pools, were evaluated in a clay loam soil in southern Alberta, Canada. A suite of laboratory-based indices were evaluated for prediction of soil N supply. The treatments were three rates (13, 39, 77 Mg  $ha^{-1}$  dry wt.) of FM or CM containing either straw or wood-chip bedding, 100 kg N ha<sup>-1</sup> as inorganic fertilizer, and an unfertilized control. Treatments were fall-applied annually for 8 years (1998–2005). Soil samples (top

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15 cm) were collected in spring 2006. The medium and high rates of organic amendment resulted in increases in  $N_0$ , and readily (Pool I) and intermediate (Pool II) mineralizable N pools in ranges of 140–355 % compared with the average of the fertilizer and control treatments. Fertilizer application had no significant effect on mineralizable N pools, but increased the mineralization rate constant  $(k)$  compared with the control. Application of FM and use of straw bedding resulted in a greater quantity of readily available and intermediate mineralizable N, and also increased the rate of N turn-over as indicated by greater values of  $k$ , compared with CM and wood-chip bedding. Among laboratory-based measures of soil N supply,  $CaCl<sub>2</sub>$ – NO<sub>3</sub> ( $r^2 = 0.84$ ) and NaHCO<sub>3</sub>-205 ( $r^2 = 0.79$ ) were strong predictors of plant N uptake (PNU). Increased soil mineralizable N did not translate into greater barley dry-matter yield or PNU. Composted beef manure and use of wood-chip bedding can be recommended as alternatives to FM and use of straw bedding for barley production is Southern Alberta.

Keywords Barley · Bedding · Compost · Fresh manure · Nitrogen mineralization · Nitrogen uptake · Soil nitrogen supply

### Introduction

Improving manure management to benefit both agricultural production and the environment requires a

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thorough understanding of the long-term effects of applied manure on active and stable soil organic nitrogen (SON) fractions. The first consideration when manure is applied as a nutrient source is usually to meet the crop N requirements in the year of application (Wen et al. [2003\)](#page-15-0). However, only a small proportion of the N in solid beef manure is in mineral form (Larney et al. [2006](#page-15-0)). In addition, the availability of N in solid beef manure is low and in some cases net immobilization may occur following manure application (Helgason et al. [2007](#page-14-0)). A better understanding of the changes in the size and quality of soil mineralizable N pools under long-term application of manure is critical in optimizing crop yield, quality, and N use efficiency to increase productivity and reduce environmental impacts.

High transportation costs often result in high rates of manure application on agricultural land near livestock operations (Freeze et al. [1999](#page-14-0)). The traditional role of manure as a nutrient source is diminished in such scenarios, and application rates may exceed that required to meet crop nutrient requirements. High application rates of fresh manure are unsustainable in the long-term, leading to adverse effects on soil (Hao and Chang [2003](#page-14-0)), water (Chang and Entz [1996](#page-14-0); Chang and Janzen [1996](#page-14-0)) and air quality (Chang et al. [1998](#page-14-0)). Consequently, the increase in the number and size of feedlots in Alberta, Canada over the past decade has resulted in many producers seeking alternative methods to land apply of raw manure to cropland used for barley and corn silage production (Miller et al. [2009](#page-15-0)).

Composting is an effective means of reducing manure volume and weight, and also increases the stability and uniformity of the manure (Rynk [1992](#page-15-0)). In addition, composting can eliminate pathogenic bacteria (Larney et al. [2003](#page-14-0)), human parasites (Van Herk et al. [2004](#page-15-0)) and weed seed viability (Larney and Blackshaw [2003\)](#page-14-0). Composting can also influence the composition and availability of N in solid beef manure (Helgason et al. [2007](#page-14-0)). Bedding is an important input at beef cattle feedlots and cereal straw is commonly used as animal bedding in Alberta, Canada. Wood residuals have been explored as alternative bedding material in recent years due to less availability and high cost of cereal straw. Differences in the C:N ratio and chemical composition of cereal straw and wood residuals can influence the availability of N in fresh and composted manure (Miller et al. [2009](#page-15-0)) and consequently influence the soil N dynamics following long-term manure application.

The size of the mineralizable N pool in soil is affected by crop management, organic amendment decomposition stage (i.e., fresh vs composted), bedding material and application rate, biotic and abiotic soil characteristics, and environmental factors such as soil temperature and water content (Griffin [2008](#page-14-0)). While single applications of manure contribute only a small amount to mineralized N in the subsequent year, the combined contributions of organic N from repeated applications can lead to a substantial increase in soil N mineralization potential (Eghball et al. [2002](#page-14-0); Flavel and Murphy [2006;](#page-14-0) Mallory and Griffin [2007](#page-15-0)). The increased soil N mineralization potential can lead to greater soil N supply and subsequently reduce the need for fertilizer N, or increase risk of excess N losses to the environment (Whalen et al. [2001](#page-15-0); Snapp and Fortuna [2003;](#page-15-0) Sharifi et al. [2011](#page-15-0)).

In contrast to soil mineral nitrogen (SMN), which is plant available and easy to quantify, the release of N from organic forms is dependent on the mineralization process (Beauchamp [1986\)](#page-14-0). History of organic amendment application can significantly change the nature and release rate of the active organic N fraction. Field-based approaches have been developed for the estimation of soil N supply (SNS) under field conditions (Hassink [1995](#page-14-0); Zebarth et al. [2005](#page-15-0)). While fieldbased approaches provide measures of the SNS to the crop, they are time consuming and require information measured at crop harvest and therefore cannot be used as a predictor of the SNS in making N fertilizer or manure recommendations (Hassink [1995;](#page-14-0) Sharifi et al. [2007b\)](#page-15-0). Laboratory-based measures of soil N mineralization that could be used as practical predictors of the SNS for crops in soil with history of organic amendment application have yet to be developed (Hassink [1995;](#page-14-0) Haney et al. [2001](#page-14-0); Sharifi et al. [2011](#page-15-0)).

The use of a standard laboratory-based method for measuring potentially mineralizable N was first proposed by Stanford and Smith [\(1972](#page-15-0)). This method requires 24 weeks aerobic incubation of the samples which makes it time consuming and not practical for routine use. Consequently, recent research has focused on the development of more rapid chemical extraction methods (St. Luce et al. [2011](#page-15-0)). Encouraging results have been reported for hot (100 $\degree$ C) KCl (Gianello and Bremner [1986a](#page-14-0)), phosphate–borate buffer at pH 11.2 (Gianello and Bremner [1986b](#page-14-0)), direct distillation with NaOH (Sharifi et al. [2009](#page-15-0)), ultraviolet absorbance of 0.01  $M$  NaHCO<sub>3</sub> extracts at 205 and 260 nm (Hong

et al. [1990](#page-14-0); Sharifi et al. [2011\)](#page-15-0), the Illinois Soil N Test for estimating amino sugar N (Williams et al. [2007](#page-15-0)), microbial biomass C or N (Carter and MacLeod [1987](#page-14-0)), and particulate organic matter C or N (Willson et al. [2001\)](#page-15-0). Few studies have attempted to quantify the effects of long-term application of manure with contrasting maturity and bedding composition on SON fractions and capacity of soil in supplying N to the crop with a wide variety of laboratory-based methods.

The objectives of this study were to: (1) assess the effects of long-term application of fresh and composted beef manure at 13, 39 and 77 Mg  $ha^{-1}$  to forage barley on soil mineralizable N and the SNS at a site located in the Southern Alberta, Canada and (2) evaluate a series of laboratory-based measures of SMN and soil N mineralization potential as predictors of SNS in the field.

#### Materials and methods

#### Site description and study design

The field experiment was conducted on a clay loam Dark Brown Chernozemic soil (Typic Haploboroll) at Lethbridge, Alberta (49°38′ N, 112°48′ W). Background soil characteristics of the experimental plots before the first treatment (fall 1998) are reported in Table 1. Climate data were obtained from the local weather station, which is  $\langle 100 \text{ m} \rangle$  from the field experiment and are reported in Table [2.](#page-3-0) Growing

Table 1 Soil chemistry at the experimental site in the fall of 1998 before the first treatment application (from Miller et al. 2004)

Parameter <sup>a</sup>	Value <sup>b</sup>
Soil mineral N (kg N $ha^{-1}$ )	$58.2 \pm 2.2$
Available P (kg ha <sup>-1</sup> )	$42.6 \pm 4.9$
Extractable K (kg ha <sup>-1</sup> )	$55.7 \pm 20.6$
pН	$8.12 \pm 0.01$
$EC$ (dS m <sup>-1</sup> )	$0.3 \pm 0.004$
$C: N$ ratio	$13 + 1$

Available N ( $NO_3-N + NH_4-N$ ) and C:N ratio of 0- to 60-cm depth; Available P, extractable K, pH and electrical conductivity (EC) of 0- to 15-cm depth;  $\frac{b}{c}$  Mean  $\pm$  1 SE. Number of samples = 56 (14 treatment plots  $\times$  4 replicates), one composite sample was collected from each block

degree days was determined from the dates of seeding and harvest, and maximum and minimum daily temperatures during the growing season.

The experiment was initiated in the fall of 1998 with annual fall application of two manure decomposition stages (FM or CM from beef cattle) with two bedding materials [unchopped barley straw (S) or wood chips (W)] and three application rates [13, 39, or 77 Mg ha<sup>-1</sup> dry matter] arranged in a factorial randomized complete block design with four replications. In addition, an unfertilized control (CON), and an inorganic fertilizer (IN) treatment consisting of 100 kg N ha<sup>-1</sup> as NH<sub>4</sub>NO<sub>3</sub> (34–0–0) plus 17 kg P  $ha^{-1}$  as triple superphosphate (0–46–0) were included. Beef manure application rates in this area range from approximately 13–57 Mg  $ha^{-1}$  (dry wt.), with a mean value of 38 Mg ha<sup>-1</sup> (Miller et al.  $2009$ ). Fertilizer recommendations for irrigated barley silage in Alberta are 50–130 kg N ha<sup>-1</sup> and 15–22 kg P ha<sup>-1</sup> (Alberta Agriculture and Rural Development [1995](#page-14-0)). The experimental unit was a plot  $6 \times 25$  m in size.

The wood-chip bedding (Sunpine Forest Products, Sundre, AB, USA) consisted of a mixture of 50 % wood chips, bark, and post peelings, and 50 % fine sawdust. The tree source of the wood chip bedding was a 4:1 mixture of lodgepole pine (Pinus contorta var. latifolia Engelm.) and white spruce [Picea glauca (Moenich) Voss]. Details of the feedlot, pen manure, and bedding material (Miller et al. [2003\)](#page-15-0), and details of the composting process (Miller et al. [2005](#page-15-0)) have been previously reported.

Manure, compost, and inorganic fertilizer were applied annually to the plots in the fall (October) of 1998 through 2005. Manure was incorporated to a depth of approximately 20 cm using an offset disc cultivator. 'Duke' barley was used in 1999–2004, and 'Kasota' was used in 2005. The crop was seeded in May at a rate of 134 kg ha<sup> $-1$ </sup> and irrigated with a sideroll system and the amount of irrigation recorded. The crop was harvested in August 2006 at the silage, softdough, or Zadock's growth stage 85, at between 60 and 70 % moisture content. The crop was harvested by cutting above-ground plant material  $(≥10$  cm) from 16  $m<sup>2</sup>$  areas from the middle of each plot with a forage harvester and total fresh weight were measured. Plant sub-samples oven-dried at 60 $\degree$ C, weighed and were fine-ground  $(\langle 150 \mu m \rangle)$  in a Cyclone plant tissue grinder (Udy Corp., Fort Collins, CO, USA) before analyses for total C and N. Total plant C and N were

Year	Precipitation		Irrig.	Avg. temp.	Total evaporation	Total sunshine	GDD
	April (mm)	May-August (mm)	May-August (mm)	May–August $(^{\circ}C)$	May-August (mm)	May-August (h)	
1998		296		-			
1999	41.5	229	229	15.0	1,084	<b>NA</b>	1,073
2000	37.4	89	330	16.5	1,235	NA	1,100
2001	44.0	65	279	17.5	1,283	636	1,066
2002	30.7	425	152	15.2	996	1,047	939
2003	68.4	123	178	16.8	1,134	1,050	1,323
2004	18.4	266	191	15.1	879	941	1,015
2005	25.5	375	152	15.4	1,040	1,048	1,091
2006	43.7	140	152	17.5	1,237	1,178	1,016
$LTM^a$	31.8	189		15.3	1,132	1,133	1,069

<span id="page-3-0"></span>Table 2 Climatic conditions, irrigation water applied, growing degree days, and seeding date of forage barley on experimental plots at Lethbridge from the time of establishment in fall of 1998 to 2006

GDD growing degree day, NA not available

<sup>a</sup> LTM, long-term (30-year) mean for April (second column) or for May to August (columns 3, 5, 7); The exception is for evaporation and GDD, where the LTM is the average from 1999 to 2006

analyzed using the Dumas automated combustion technique (McGill and Fiqueiredo [1993\)](#page-15-0) using a CNS analyzer (Carla Erba, Milan, Italy). Total plant N uptake (PNU) was calculated by multiplying total plant dry weight and plant N concentration.

# Amendment analysis

Fresh or composted manure was sampled from piles before land application. Twenty random shovelfuls of material were taken from each pile, composited into one sample, and then between 5 and 36 sub-samples were taken from the composite pile for chemical analyses. All manure samples were oven-dried at 60  $\degree$ C, ground to pass a 2-mm sieve, and then extracted for chemical analyses. The exception was  $NO<sub>3</sub>-N$  and  $NH<sub>4</sub>-N$ , where extractions were conducted on fresh manure samples the day of sampling. The  $NO<sub>3</sub>-N$  and  $NH<sub>4</sub>-N$  in the manure were extracted using a 1:20 ratio of 10 g of manure and 200 mL of 2 M KCl after shaking at low speed for 1 h. The  $NH_4-N$  was determined using the Berthelot reaction on the autoanalyzer (Technicon Industrial Systems, 1978). Electrical conductivity (EC) and pH in manure were determined on 1:5 manure and water extracts (20 g manure and 100 mL distilled water) after shaking at low speed for 1 h. Samples for total C and N analyses

were also oven-dried at  $60^{\circ}$ C and finely ground to pass a 150-um sieve. Organic C and N were determined using Dumas automated combustion technique using a CNS analyzer (Carla Erba, Milan, Italy). Details of extraction methods and chemical analysis have been previously reported by Miller et al. ([2003](#page-15-0)). Chemical analysis and cumulative total N applied in eight years of fresh and composted beef cattle manure application are reported by Miller et al. ([2009\)](#page-15-0) and summarized in Tables [3](#page-4-0) and [4](#page-5-0).

# Soil sampling and analysis

Composite soil samples were collected from 0- to 15-cm depth of each plot in spring of 2006 before seeding. Soils were air-dried and sieved  $\leq$ 2-mm) before analysis. Soil texture (pipette method following removal of carbonates and organic matter; Gee and Bauder [1986\)](#page-14-0) and soil pH (1:1 soil:water suspension) were determined. Electrical conductivity (EC) of soil samples was determined by measuring the electrical resistance of 1:1 soil water suspension (Table [4](#page-5-0)). The SOC and SON were measured by the dry combustion method following carbonate removal using a LECO CNS-1000 analyzer (Table [6\)](#page-9-0). A modification of the Stanford and Smith [\(1972](#page-15-0)) long-term aerobic incubation procedure was used to measure potentially

Parameter <sup>a</sup>	FM-straw	$CM$ -straw	FM-wood	CM-wood	
Dry matter $(g \text{ kg}^{-1})$	$598 \pm 94^{\rm b}$	$671 \pm 97$	$598 \pm 95$	$721 \pm 97$	
pH	$8.3 \pm 0.1$	$8.0 \pm 0.1$	$7.6 \pm 0.2$	$7.5 \pm 0.2$	
$EC$ (dS m <sup>-1</sup> )	$9.2 \pm 1.1$	$9.6 \pm 0.4$	$6.7 \pm 0.4$	$5.8 \pm 0.3$	
Total C $(g \text{ kg}^{-1})$	$239 \pm 23.5$	$191 \pm 15.0$	$340 \pm 6.9$	$269 \pm 21.0$	
Total N $(g \text{ kg}^{-1})$	$16.4 \pm 0.6$	$16.4 \pm 0.7$	$15.8 \pm 0.8$	$14.3 \pm 0.8$	
$C: N$ ratio	$14.6 \pm 1.4$	$11.5 \pm 0.5$	$22.2 \pm 1.0$	$18.4 \pm 0.8$	
$NH_4$ -N (mg kg <sup>-1</sup> )	$1,145 \pm 240$	$434 \pm 169$	$947 \pm 158$	$471 \pm 193$	
$NO_3-N$ (mg kg <sup>-1</sup> )	$274 \pm 82.7$	$323 \pm 93.6$	$162 \pm 27.9$	$250 \pm 45.9$	

<span id="page-4-0"></span>Table 3 Chemical analysis of fresh (FM) and composted (CM) manure containing straw or wood-chip bedding (from Miller et al. [2009\)](#page-15-0)

EC electrical conductivity

<sup>a</sup> All chemical analysis expressed on a dry weight basis. Each number is average value over 9 years (1999–2007), and on average 15 samples of each amendment were taken each year  $(N = 135)$ 

Mean  $\pm$  1 SE

mineralizable soil N  $(N_0)$  (Curtin and Campbell [2008](#page-14-0)). Soil samples (30 g) from 0- to 15-cm were mixed with sand (60 g), packed into leaching tubes, and wetted to 55 % of the soils' water-holding capacity by leaching followed by suction  $({\sim}10 \text{ kPa})$  and incubated at  $25 \text{ °C}$  for 44 weeks. Therefore, soils' moisture contents were kept at 55 % of the soils' water-holding capacity during the incubation period. The soils were leached periodically (every 2 weeks for the first 12 weeks and every 4 weeks thereafter) with  $0.01$  *M* CaCl<sub>2</sub> followed by a zero-N nutrient solution (Curtin and Campbell [2008](#page-14-0)). Leachates were analyzed for concentrations of  $NH_4$ -N and  $NO_3$ -N colorimetrically. The N mineralized in the first 2 weeks period was not used for the curve fitting procedure because this represents the initial flush of mineralization upon rewetting. The following first-order kinetic model was fitted to data using the Marquardt iteration method:

$$
N_{\min} = N_0[1 - \exp(-kt)] \tag{1}
$$

where  $N_{\text{min}}$  is the net cumulative amount of N mineralized at time, t,  $N_0$  is potentially mineralizable N, and  $k$  is the mineralization rate coefficient (Curtin and Campbell [2008](#page-14-0)). Three different pools of mineralizable N based on long-term aerobic incubation were calculated (Sharifi et al. [2007a](#page-15-0)):

[Pool I] The flush in mineral N which occurs in the first 2 weeks period following rewetting. This pool represents a labile mineralizable organic-N pool.

[Pool II] Cumulative amount of N mineralized between weeks 2 and 44 and represents an intermediate pool of mineralizable organic-N.

[Pool III] The amount of SON that was potentially mineralizable, but did not mineralize during the incubation period and was calculated by difference between  $N_0$  predicted from curve fitting and the cumulative amount of N mineralized between weeks 2 and 44 (Pool II). This represents a stable pool of mineralizable organic-N.

The KCl extractable  $NO_3-N$  and  $NH_4-N$  (KCl– $NO_3$ ) and  $KCl-NH_4$ ) were determined using extraction with 1.7 M KCl (20 g air dry soil:100 mL extractent) at room temperature and analyzed colorimetrically using a Technicon TRAACS 800 auto-analyzer (Zebarth and Milburn [2003](#page-15-0)). Hot KCl extractable  $NH_4-N$  (HKCl– NH4) was determined by heating 9.0 g soil with 60 mL 2 M KCl on a digestion block set at 100 °C for 4 h followed by extraction and analysis for  $NH_4$ -N as described above (Gianello and Bremner [1986a\)](#page-14-0). The ultraviolet absorbance of the  $0.01$  M NaHCO<sub>3</sub> extract at 205 and 260 nm (NaHCO<sub>3</sub>-205 and NaHCO<sub>3</sub>-260) was determined as described by Serna and Pomares [\(1992](#page-15-0)). Briefly, 2.5 g of soil were shaken in 50 mL of  $0.01$  *M* NaHCO<sub>3</sub> for 15 min in 125 mL Erlenmeyer flasks. The suspension was suction filtered through Whatman no. 42 filter paper and the ultraviolet absorbance of the extract was measured at 205 and 260 nm with an ultraviolet spectrometer (Beckman DU 530 UV/VIS Spectrophotometer, Beckman



<span id="page-5-0"></span>



d

Source of the data for TDM and PNU is Miller et al. [\(2009](#page-15-0))

Source of the data for TDM and PNU is Miller et al. (2009)

Coulter, Fullerton, CA, USA). Illinois Soil N Test (ISNT) was determined using the direct diffusion method described by Khan et al. [\(2001](#page-14-0)). Phosphate– borate buffer extractable N (PBN) was determined by the method of Gianello and Bremner ([1986b](#page-14-0)). The 4.00 g soil sample, with 40 mL of phosphate–borate buffer (pH 11.2), was direct steam distilled for 8 min to obtain 40 mL of distillate. The  $NH_4$ -N was determined in the distillate by back-titration of boric acid with standard  $0.01$  M H<sub>2</sub>SO<sub>4</sub>. The NaOH direct-distilled N was measured by steam distillation of a 5 g soil sample with 20 mL of 12.5 M NaOH for 10 min, after which the  $NH_4$ -N concentration in the distillate was backtitrated with standard  $0.01 M H_2SO_4$  (Sharifi et al. [2009\)](#page-15-0). The chloroform fumigation extractable C was determined as an index of microbial biomass C (MBC) (Voroney et al. [2007](#page-15-0)). The POM-C and POM-N were determined by passing a 25 g dispersed air-dried soil sample through a 53 µm sieve (Gregorich and Beare [2008\)](#page-14-0). Retained sand and macroorganic matter were dried and weighed. Carbon and N concentrations were then determined on this retained fraction by dry combustion using a LECO CNS-1000 and the masses of C and N per gram of air-dry soil were calculated as POM-C and POM-N, respectively. All measurement units were converted to units of kg N  $ha^{-1}$  using soil bulk density of each plot measured by the soil core method (Blake and Hartge [1986](#page-14-0)).

# Statistical analysis

Two MIXED model analyses (Littell et al. [1998](#page-15-0)) were conducted in this study. The first analysis was conducted to determine the effect of the main treatment factors which were manure decomposition stage, bedding material, and manure application rate on the dependent variables. For this analysis, the control and inorganic fertilizer treatments were omitted as there was only one level of each of these factors. Main treatment and interaction effects were tested using least-squares means (LSM). A probability level of  $P \leq 0.05$  was considered significant for F statistic values and LSM comparisons.

A second MIXED model analysis was conducted to determine the effect of all 14 individual treatments, including the control and inorganic fertilizer treatment, on the dependent variables. The 14 treatments were compared after 8 years (1998–2006) of amendment and fertilizer application. Contrast statement was <span id="page-7-0"></span>also used in this analysis to compare the various individual treatments (e.g., control vs. FM) and combinations of treatments (e.g., control vs. FM and CM). A probability level of  $P \le 0.05$  was considered significant for mean and contrast comparisons.

The dry-matter yield (TDM) and PNU in the harvested forage barley were used as field-based indices of SNS (Zebarth et al. [2005](#page-15-0)). Correlations between laboratory-based and field-based indices of SNS were determined using Spearman's rank correlation (Proc CORR, Spearman) procedure. The significant correlations ( $P \le 0.05$ ) (positive or negative) were determined to be relevant for further consideration using linear and non-linear regression analyses. Correlation and regression analysis in SAS were conducted using the CORR procedure and REG procedure, respectively. A probability level of  $P \leq 0.05$  was considered significant regressions.

#### Results and discussion

Laboratory-based measures of soil mineralizable nitrogen

The parameter  $N_0$  is a measure of the active soil N fraction (Drinkwater et al. [1996](#page-14-0)) and was considered as the standard measure of N mineralization potential (Curtin and Campbell [2008](#page-14-0)). The  $N_0$  represents the sum of mineralizable N Pools II and III (Sharifi et al. [2007a](#page-15-0)). The  $N<sub>0</sub>$  ranged from 352 to 1,628 mg N kg<sup>-1</sup> soil (Table [4](#page-5-0)). These values represented an average of 9.3 % of SON, although the  $N_0$ /SON ratio was 27 % greater in straw bedding compared with woodchip bedding.

The  $N_0$  was significantly affected by manure decomposition stage, rate of manure application and their interaction. There was no significant effect of bedding on  $N_0$ . Greater average  $N_0$  values were observed in CM compared with FM, and  $N_0$  values generally increased with increased manure application rate (Table [4](#page-5-0)). Values of  $N_0$  were more responsive to manure application rate in CM compared with FM (Fig. 1a). Addition of each Mg  $ha^{-1}$  of FM resulted in a 1.3–8.1 kg N ha<sup>-1</sup> increase in  $N_0$ , whereas addition of each Mg ha<sup>-1</sup> of CM resulted in a 13-18 kg N ha<sup>-1</sup> increase in  $N_0$ . The  $N_0$  values in CON and IN were not significantly different (Tables [4,](#page-5-0) [5](#page-8-0)) and were lower than all manured treatments.



Fig. 1 Effect of manure decomposition stage and application rates on potentially mineralizable N  $(N<sub>0</sub>)$  and the intermediate mineralizable N pool (Pool lI) (a), and manure and bedding material type on KCl extractable  $NO<sub>3</sub>-N$  (KCl–NO<sub>3</sub>) and total soil organic N (b). Bars represent standard errors

The direct comparison of  $N<sub>0</sub>$  values calculated in this study against previously published values is difficult due to differences in methodologies used. However, the values we obtained are within the range of values previously reported by Simard and N'Day-egamiye ([1993;](#page-15-0) 232-1,976 kg N ha<sup>-1</sup>, assuming a soil bulk density of 1.2 Mg  $m^{-3}$ ; 55.4 weeks incubation at 20  $^{\circ}$ C) for 20 meadow soils in Quebec, Canada, but were greater than those reported by Sharifi et al. (Sharifi et al. [2008](#page-15-0); 101-284 kg N ha<sup>-1</sup>) for a longterm beef manure amended potato soil in Maine, USA; by Sharifi et al.  $(2011; 182-480 \text{ kg N ha}^{-1})$  $(2011; 182-480 \text{ kg N ha}^{-1})$  for a long-term beef manure amended forage grass in Eastern Canada; by Sbih et al. ([2003](#page-15-0); 158–423 kg N ha<sup>-1</sup>, assuming a soil bulk density of

<span id="page-8-0"></span>Table 5 Contrast for differences among various combination of all 14 treatments for selected parameters

Treatment <sup>a</sup>	$N_O^{\rm b}$	$\boldsymbol{k}$	Pool I	Pool II	$CaCl2-NO3$	$NaHCO3 - 205$	TDM	<b>PNU</b>
CON versus CM-straw								
13	<b>NS</b>	<b>NS</b>	<b>NS</b>	<b>NS</b>	$\ast$	***	***	***
39	$**$	<b>NS</b>	***	***	***	***	***	***
77	***	<b>NS</b>	***	***	***	***	***	***
CON versus CM-wood								
13	<b>NS</b>	<b>NS</b>	∗	<b>NS</b>	<b>NS</b>	***	***	<b>NS</b>
39	<b>NS</b>	<b>NS</b>	$\ast$	$\ast$	<b>NS</b>	***	***	***
77	***.	<b>NS</b>	***	***	***	***	***	***
CON versus FM-straw								
13	<b>NS</b>	<b>NS</b>	<b>NS</b>	$\ast$	***	***	***	***
39	<b>NS</b>	***	NS	***	***	***	***	***
77	$\ast\ast$	*	***	***	***	***	$\ast$	***
CON versus FM-wood								
13	<b>NS</b>	<b>NS</b>	NS	<b>NS</b>	<b>NS</b>	***	***	NS.
39	<b>NS</b>	<b>NS</b>	***	*	***	***	***	***
77	$***$	NS	***	***	***	***	***	***
CON versus IN	<b>NS</b>	***	<b>NS</b>	<b>NS</b>	***	***	***	***

NS not significant at 0.05 probability level

<sup>a</sup> FM, fresh manure; CM, composted manure; application rates: 13, 39, and 77 Mg ha<sup> $-1$ </sup> dry weight basis; CON, control with no manure and no fertilizer; IN, inorganic fertilizer consist of 100 kg N ha<sup>-1</sup> and 17 kg P ha<sup>-1</sup>

 $b_{N_0}$  = potentially mineralizable N;  $k =$  mineralization rate coefficient; Pool I = cumulative amount of N mineralized in the first 2 weeks following rewetting; Pool II = cumulative amount of N mineralized between 2 and 44 weeks; Pool III =  $N_0$  minus Pool II; NaHCO<sub>3</sub>-205 and -260 = ultraviolet absorbance of 0.01 M NaHCO<sub>3</sub> extract at 205 and 260 nm, respectively.; TDM = total barley above  $=$  -ground dry-matter yield; PNU  $=$  total barley N uptake in above-ground plant

1.2 Mg m<sup>-3</sup>; 56 weeks incubation at 25 °C) for 34 dairy manure amended meadow soils in Quebec, Canada; and by Griffin and Laine (Griffin and Laine [1983;](#page-14-0) 261-788 kg N ha<sup>-1</sup>, assuming a soil bulk density of 1.2 Mg  $m^{-3}$ ; 40 weeks incubation at 35  $\degree$ C) for 17 soils amended with beef manure, poultry manure and sewage sludge in USA. The greater values of  $N_0$  in this study compared with most of the previous studies could be related to cumulative effect of the long-term annual application of organic amendments at high rates in combination with high inherent SON in the Chernozemic soils in this study. The observed ratio of  $N_0$  to SON was in the range (9–10 %) reported by Sharifi et al. ([2008;](#page-15-0) [2011](#page-15-0)) for long-term beef manure amended soils in northeastern USA and eastern Canada. As the cumulative amount of added total N in CM and FM was comparable (Table [4\)](#page-5-0), the more rapid increase in  $N_0$  with increase in application rates of CM compared with FM can be attributed to more recalcitrant forms of organic N in CM than in FM.

The k values ranged from 0.035 to 0.094 week<sup>-1</sup> (Table [4\)](#page-5-0). This range is similar to the range of values reported by Curtin and Wen  $(1999; 0.025-0.178 \text{ week}^{-1})$  $(1999; 0.025-0.178 \text{ week}^{-1})$ , Sharifi et al.  $(2008; 0.013-0.136$  $(2008; 0.013-0.136$  week<sup>-1</sup>), and Sharifi et al.  $(2011; 0.023-0.107$  $(2011; 0.023-0.107$  $(2011; 0.023-0.107$  week<sup>-1</sup>). In contrast, Stanford and Smith  $(1972)$  $(1972)$  found that the value of k was nearly constant across soils at  $0.050 \pm 0.009$  week<sup>-1</sup>.

There were significant manure decomposition stage  $\times$  bedding and manure decomposition stage  $\times$ manure application rate on k values (Table [4](#page-5-0)). Bedding type had no effect on  $k$  at different rates of CM with straw bedding or wood-chip bedding, however, medium and high rates of FM with straw bedding resulted in significantly  $(p<0.1)$  higher k values compared with the control. Values of  $k$  were 54 % greater for straw compared with wood-chip bedding for FM, whereas for CM,  $k$  values were 19 % greater for straw compared with wood-chip bedding. The k values in the IN treatment  $(0.094 \text{ week}^{-1})$  was numerically greater than for all other treatments, and

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the k values in the control treatment  $(0.036 \text{ week}^{-1})$ were numerically lower than almost all other treatments. The lower values of  $k$  for wood-chip than for straw bedding likely reflects the greater content of C in the form of lignin in wood-chips  $(\sim 280 \text{ g kg}^{-1})$ compared with straw ( $\sim$ 110 g kg<sup>-1</sup>) (Larney et al. [2008\)](#page-15-0). Decomposition and N release generally occurred faster for residues with lower C/N ratios and lignin and polyphenol contents (Seneviratne [2000\)](#page-15-0). In a study of green manure crops, leaves decomposed five times faster than stems, decomposition was closely related to cell wall content, and N release was most dependent on the lignin/N ratio (Cobo et al. [2002\)](#page-14-0). Mendham et al. [\(2004](#page-15-0)) reported that biochemical characteristics of added organic residues other than the C/N ratio had the main influence on net N mineralization rates.

Pool I ranged from 25 to 157 mg N  $kg^{-1}$  soil (Table [4](#page-5-0)). Pool I was 28 % greater for CM compared with FM, but was not significantly affected by bedding type. Pool I increased linearly with manure application rate with the average increase of 0.80 kg N  $ha^{-1}$  per Mg of organic amendment application. The lowest values of Pool I were measured for the CON and IN treatments (Table [4\)](#page-5-0).

Pool II ranged from 294 to 1,399 kg N  $ha^{-1}$ (Table [4](#page-5-0)) and constituted 76–99 % of  $N_0$ , while Pool III constituted 1–24 % of  $N_0$ , averaged across all treatments. Pool II was 18 % greater for CM compared with FM, and 21 % greater for straw compared with wood-chip bedding. There was a significant manure decomposition stage  $\times$  rate interaction on Pool II where Pool II increased more rapidly with increasing application rate for CM than for FM (Fig. [1a](#page-7-0)).

Pool III ranged from 0 to 340 kg N  $ha^{-1}$  and had the greatest CV values of any mineralizable N parameter (Table [4](#page-5-0)). Pool III was three times greater for CM compared with FM, but was not significantly affected by bedding material. There was a significant manure decomposition stage  $\times$  rate interaction on Pool III, where Pool III increased with increasing rate of CM but was not affected by rate of FM.

Previous studies observed an increase in  $N_0$  as a result of long-term application of an organic amend-ment (Flavel and Murphy [2006](#page-14-0); Griffin [2008](#page-14-0)), where greater amendment application rates resulted in proportionally greater values of  $N<sub>0</sub>$  (Whalen et al. [2001](#page-15-0)). Mallory and Griffin ([2007](#page-15-0)) reported a 100 % increase in net N mineralization by history of solid beef manure application in a potato–barley rotation using a cumulative measurement of mineral N during a 282 day aerobic incubation. Similarly, Willson et al. ([2001\)](#page-15-0) found that  $N_0$  was increased by the addition of dairy manure compost and Sharifi et al.  $(2011)$  $(2011)$  found that  $N_0$ was increased by long-term application of semi-solid beef manure. Solid beef manure commonly contains organic N compounds with high resistance to mineralization (Beauchamp [1986\)](#page-14-0) and this suggests that annual application of organic amendments may build up the mineralizable N pools coupled with an increased risk of N leakage to the environment (Sharifi et al. [2011\)](#page-15-0). The build up of mineralizable N pools was enhanced in the current study by the composting process.

It is useful to note that the long-term manure applications in this semi-arid environment had a significant effect on soil EC (Table [4\)](#page-5-0). Soil EC increased with increasing application rate, but was not significantly affected by manure decomposition stage or by bedding material.

The increase in EC of the soil as result of higher application rates of FM or CM (Table [4](#page-5-0)) resulted in lower MBC values (Table [6](#page-9-0)), which consequently could have impacted N mineralization and nitrification. The soil N mineralizable pools and mineralization rate can also be indirectly influenced by a history of amendment application through effects on other edaphic factors such as soil pH, soil physical properties and soil microbiological communities (Mallory and Griffin [2007](#page-15-0)).

## Laboratory-based measures of nitrogen availability

The KCl-NO<sub>3</sub> ranged from 31 to 305 kg N ha<sup>-1</sup> (average of 126 kg N ha<sup>-1</sup>) and KCl-NH<sub>4</sub> ranged from 3 to 39 kg N  $ha^{-1}$  (average of 12 kg N  $ha^{-1}$ ) across all treatments (Table  $6$ ). The KCl–NO<sub>3</sub> and KCl–NH<sub>4</sub>-N constituted 1.32 and 0.12 % of the average SON, respectively. The SMN ranged from 33 to 344 kg N  $ha^{-1}$  (average 139 kg N  $ha^{-1}$ ) and consisted of 9 % of NH<sub>4</sub>-N and 91 % NO<sub>3</sub>-N. There was a significant manure decomposition stage  $\times$  bedding interaction on  $KCl-NO<sub>3</sub>$  where values of  $KCl-$ NO3 were greater and more responsive to bedding for FM than for CM. The  $KCl-NO<sub>3</sub>$  also increased with increased manure application rate. The KCl–NH4 content was significantly affected by interaction

 **(TDM)**

**0**

**0**

**1**

**2**

**3 4**

**TDM (kg N ha-1)**

TDM (kg N ha<sup>-1</sup>)

**5 6**

**7**

**1**

**2 3**

**4**

**TDM (kg N ha-1)**

 $TM$  (kg  $N$  ha<sup>-1</sup>)

**5**

**6 7**

 $y = -7E - 05x^2 + 0.017x + 4.4438$  $R^2 = 0.1298$ 

m

'n

 $y = -0.0129x^{2} - 0.055x + 105.73$  $R^2 = 0.6738$ 

**y = 0.1732x + 83.105**  $R^2 = 0.2696$ 

<span id="page-11-0"></span>

Fig. 2 Relationships between total barley N uptake (PNU) or total barley dry-matter yield (TDM) and potentially mineralizable N  $(N_0)$ , the readily mineralizable N pool (Pool I), the

intermediate mineralizable N pool (Pool lI) and the stable mineralizable N pool (Pool III) to  $N_0$  ratio

Pool III/N<sub>0</sub>

between manure decomposition stage and application rate. The  $KCl-NH<sub>4</sub>$  content responded linearly to increase in CM rates, however this response was quadratic or linear plateau for FM. In most cases values of  $KCl-NH<sub>4</sub>$  were small in comparison with KCl–NO<sub>3</sub>. The hot KCl–NH<sub>4</sub> was on average 5.4 times greater that of KCl–NH4, with values ranging from 13 to 183 kg N ha<sup>-1</sup> (Table [6\)](#page-9-0) and accounted for 0.33–1.1 % of total SON. The hot KCl–NH<sub>4</sub> was significantly greater for wood-chip than for straw bedding, indicating that wood-chips are an effective absorbent of N fraction, which may become available after strong chemical treatment. Increasing manure application rate also increased hot  $KCl-NH<sub>4</sub>$  whereas there was no effect of manure decomposition stage on hot KCl–NH<sub>4</sub>.

The  $CaCl<sub>2</sub>-NO<sub>3</sub>$  values ranged from 31 to 235 kg N  $ha^{-1}$  and were found to be similar to that of KCl–NO<sub>3</sub>. The UV absorbance of 0.01  $M$  NaHCO<sub>3</sub> extracts ranged from 1.31 to 3.53 at 205 nm and from 0.29 to 1.96 at 260 nm (Table  $6$ ). These values are slightly greater than the values reported by Hong et al.

<span id="page-12-0"></span>

Fig. 3 Relationships between total barley N uptake (PNU) or total barley dry-matter yield (TDM) and CaCl<sub>2</sub> extactable  $NO<sub>3</sub>$ -N (CaCl<sub>2</sub>–NO<sub>3</sub>), the UV absorbance of 0.01 *M* NaHCO<sub>3</sub>

[\(1990](#page-14-0); 0.58–2.49 at 205 nm and 0.20–0.44 at 260 nm), by Serna and Pomares [\(1992](#page-15-0); 0.51–2.17 at 205 and 0.24–0.86 at 260 nm), and by Sharifi et al.  $(2011;$  $(2011;$ 0.71–2.58 at 205 nm and 0.38–1.35 at 260 nm). The  $NaHCO<sub>3</sub> - 205$  was greater for straw than for wood-chip bedding and increased with manure application rate, but was not affected by manure decomposition stage. In comparison,  $NaHCO<sub>3</sub>$ -260 increased with manure application rate, but was not affected by manure decomposition stage or bedding.

Values of ISNT-N were significantly greater for CM than FM, greater for wood-chip than for straw

extracts at 205 nm, the readily mineralizable N pool (Pool I) plus  $CaCl<sub>2</sub>–NO<sub>3</sub>$ , and soil electrical conductivity (EC)

bedding, and increased with manure application rate (Table [6](#page-9-0)). In comparison, the NaOH-DD was greater for wood-chip than straw bedding, but was not affected by manure decomposition stage. There was a manure decomposition stage  $\times$  rate interaction on NaOH-DD where values of NaOH-DD increased more rapidly with increasing application rate for CM than for FM. In contrast, there was a significant response of PBN to manure application rate only.

The MBC ranged from 432 to 1,723 kg C ha<sup>-1</sup> with an average value of 1,01[6](#page-9-0) kg C ha<sup>-1</sup> (Table 6) and accounted for 0.4–2.3 % of total SOC. This is within the

range of values reported by Sharifi et al. ([2011](#page-15-0); 333–2,010 kg C  $\text{ha}^{-1}$ ) for a soil under forage grass with long term history of beef manure application. There was a significant manure decomposition stage  $\times$  rate  $\times$  bedding interaction on MBC. Maximum values of MBC occurred at rates 13 or 39 Mg  $ha^{-1}$ depending on manure decomposition stage and bedding, and were generally lower for FM with straw bedding than for other treatment combinations.

The POMC ranged from 8 to 128 Mg  $ha^{-1}$ , and was equivalent to 24–[6](#page-9-0)8 % of total SOC (Table 6). Similarly, the POMN ranged from 0.4 to 8.1 Mg  $ha^{-1}$ , and was equivalent to 7–55 % of total SON. The POMN was the largest of the organic N fractions evaluated. The C:N ratio of POM was wider (average 16, range 11–21) than that of the whole soils (average 10, range 6–13), reflecting the dominant influence of C through bedding (straw or woodchip) on this pool (Christensen [1992](#page-14-0)). The POMC was greater for woodchip than for straw bedding and increased with manure application, but was not affected by manure decomposition stage.

# Relationships between field-based indices and laboratory-based measures of nitrogen supply

The field based indices of SNS (i.e., TDM and PNU) are reported and discussed by Miller et al. [\(2009](#page-15-0)). Correlation coefficients were calculated between laboratory-based measures of soil N availability and TDM or PNU as field-based indices of N supply. Among laboratory-based measures of soil N supply,  $CaCl<sub>2</sub>-NO<sub>3</sub>$  (r = 0.78), NaHCO<sub>3</sub>-205 (r = 0.86), a combination of Pool I-NO<sub>3</sub> and CaCl<sub>2</sub>-NO<sub>3</sub>  $(r = 0.76)$ , Pool III: $N<sub>0</sub>$  ratio  $(r = -0.79)$  and EC  $(r = 0.61)$  were strongly associated with PNU, and NaHCO<sub>3</sub>-205 (r = 0.44), EC (r = 0.35) and CaCl<sub>2</sub>- $NO<sub>3</sub>$  (r = 0.19) were associated with TDM. Linear correlation coefficients of measures of N availability with TDM were low due to lack of a linear relationship. The  $N_0$ , Pool I and Pool II were weakly correlated with PNU  $(r = 0.50, 0.52, \text{ and } 0.62, \text{ respectively}).$ There was no strong correlation between ISNT, MBC, NaOH-DD, POMC, POMN, SOC, SON and Hot KCl– NH4 either with PNU or TDM. Plots of PNU and TDM against the most successful laboratory-based measures of N availability were used to identify the suitability of using these parameters for prediction of N supply in the field (Figs. [2,](#page-11-0) [3](#page-12-0)). The CaCl<sub>2</sub>–NO<sub>3</sub> and NaHCO<sub>3</sub> were good predictors of PNU and predicted 84 and 79 % of the variability in this parameter, respectively. Multi-variable regression and stepwise approaches were tested but no improvement in the relationships was achieved. The superiority of mineral N based indicator to N mineralization based indicators in predicting PNU and TDM can be attributed to large quantity of soil mineral N in soil before planting. A portion of this mineral N is as a result of carryover from previous growing season. Therefore, a great proportion of SNS in this soil was comprised of soil mineral N.

Similar to our findings, in a non-amended field potato experiments in New Brunswick and PEI, Canada and Maine, USA, Sharifi et al. ([2007b\)](#page-15-0) reported that pre-plant mineral N at 0–30 cm soil depth and  $NaHCO<sub>3</sub>$ -205 were the best predictors of field-based indices of SNS.

#### **Conclusions**

Eight years history of amendment application increased soil mineralizable N pools, where greater rates resulted in greater soil mineralizable N. Manure decomposition stage  $(FM < CM)$  and application rates ( $L \ll M \ll H$ ) were the dominant factors affecting quantity of active soil organic N fraction. Application of FM and use of straw bedding resulted in a greater quantity of readily available and intermediate mineralizable N, and also increased the rate of N turnover as indicated by greater values of  $k$ , compared with CM and wood-chip bedding. Composted manure contributed more to the intermediate and stable mineralizable N pools and consequently was more effective in accumulating organic N in soil. The bedding type significantly impacted only  $k$  and Pool II among potentially mineralizable N parameters. Straw bedding resulted in 17 % greater values for Pool II compared with wood-chip bedding. There was an interaction between decomposition stage and bedding type or rate of application on  $k$ , where only medium and high rates of FM with straw bedding resulted in higher k values compared with control. Greater k values were measured in the inorganic fertilizer than in the control treatment.

The relationship between mineralizable N indices and barley TDM or PNU was polynomial. Increased soil mineralizable N in medium and high application rates <span id="page-14-0"></span>(39 and 77 Mg  $ha^{-1}$  dry matter basis) did not translate into greater barley yield or N uptake due to abundant soil N supply and high soil EC (close to crop tolerance threshold). Barley yield and N uptake were similar in fresh manure and composted manure with straw or woodchip bedding. High soil EC also resulted in reduced MBC. Among tested laboratory-based measures of N availability,  $CaCl<sub>2</sub>–NO<sub>3</sub>$ , and NaHCO<sub>3</sub> were the superior predictors of PNU and predicted 84 and 79 % of the variability in this parameter, respectively. Composted beef manure and wood-chip bedding can be recommended as alternatives to FM and straw bedding for barley production is Southern Alberta. Low rate of annual application would be appropriate due to inherent high SNS in Dark Brown Chernozemic soils of this region. Application of fresh or composted manure in semiarid region need to be tailored to crop N and P demand with close monitoring of soil EC.

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