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Mineralizable Nitrogen Responds Differently to Manure Type in Contrasting Soil Textures

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Manuring soil alters mineralizable N pools and organic matter fractions, but the magnitude is manure-type and soil-texture specific, complicating prediction of N mineralization. Our objective was to determine the responses of residual soil mineralizable N parameters to manure-type and evaluate their relationships to initial organic C and N fractions, C/N ratios, and mineral N concentrations in sandy loam and silty clay soils after three annual spring applications of manure. Manure types were liquid swine manure (LSM), liquid dairy cattle manure (LCM), or solid poultry manure (SPM), all applied at 90 kg available N ha^{-1} yr^{-1}. Mineral fertilizer (NPK) and a zero-N control (CTL) were also included. Composite soil samples collected (0- to 20-cm depth) before manure application were aerobically incubated at 25°C for 48 wk. Both soils mineralized N linearly over 48 wk (r^2 = 0.96–0.99) and the silty clay soil did not converge to nonlinear, first-order kinetics. Pool I (N mineralized in first 2 wk) was the only mineralizable N pool affected by manure-type, which was depleted by SPM in the sandy loam and increased by LCM in the silty clay. Salt extractable organic N was significantly correlated to Pool I in both soil textures. Only Pool I was significantly correlated with N mineralized over 48 wk in the sandy loam and silty clay soils (r = 0.92 and 0.64, respectively). Overall, readily mineralizable N (Pool I) was the most sensitive and robust indicator of mineralizable N after three annual manure applications to agricultural soils from a humid temperate region.

Abbreviations: KCl-NH$_4$, extractable ammonium nitrogen with 2 *M* KCl; KCl-NO$_3$, extractable nitrate nitrogen with 2 *M* KCl; KCl-Total, KCl-NH$_4$ + KCl-NO$_3$; CaCl$_2$–NH$_4$, extractable ammonium nitrogen with 0.01 *M* CaCl$_2$; CaCl$_2$–NO$_3$, extractable nitrate nitrogen with 0.01 *M* CaCl$_2$; CaCl$_2$–Total, CaCl$_2$–NH$_4$ + CaCl$_2$–NO$_3$; WEON, water extractable organic nitrogen; WEOC, water extractable organic carbon; WEMN, water extractable mineral nitrogen; Hot-WEON, hot-water extractable organic nitrogen sequential to WEON extraction; Hot-WEOC, hot-water extractable organic carbon sequential to WEOC extraction; Hot-WEMN, hot-water extractable mineral nitrogen sequential to WEMN extraction; Post hot-WEON-NH$_4$, extractable ammonium nitrogen with 2 *M* KCl sequential to hot-WEON extraction; SEON, salt extractable organic nitrogen with 0.5 *M* K$_2$SO$_4$; SEOC, salt extractable organic carbon with 0.5 *M* K$_2$SO$_4$; MBN, microbial biomass nitrogen; MBC, microbial biomass carbon; POMN, particulate organic matter nitrogen; POMC, particulate organic matter carbon.

Manure is a valuable source of N for crop production, but a large portion of the N input from manure is in organic N compounds that must be mineralized to plant-available forms to have N fertilizer value. The quantity of organic N that is mineralizable in the year manure is applied and subsequent growing seasons is not easily estimated due to the heterogeneity of manure physicochemical composition, and interactions between the N mineralization process and soil and environmental conditions (Beauchamp, 1986; Chadwick et al., 2000; Eghball et al., 2002; Van Kessel and Reeves, 2002). In manure-amended soils the fraction of organic N that does not mineralize in the year of application, cumulates and can have a residual N fertility effect (Lund and Doss, 1980; Schröder, 2005; Mallory and Griffin,

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2007). However, the magnitude of the residual effect depends on an interaction between manure type and soil texture (Webb et al., 2013). Further insight into the response of residual mineralizable N pools to manure type and soil texture could help reveal mechanisms controlling residual N fertility. Robust indicators are needed to predict residual N fertility in soils with manuring histories to optimize supplemental N fertilizer recommendations and minimize N loss to the environment.

The potentially mineralizable N (N_0) incubation is a lab-based method that estimates the mineral N concentration that could become plant-available and the N mineralization rate constant (k) of a soil (Stanford and Smith, 1972). In soils with an 8- to 14-yr history of manure application, N_0 represented 9 to 10% of the soil organic N (Sharifi et al., 2008, 2011, 2014). For example, 10 yr of semi-solid beef cattle manure applied at rates between 75 and 300 kg N ha^{-1} led to N_0 values of 326 to 480 kg N ha^{-1} in a silt loam and 202 to 304 kg N ha^{-1} in a sandy clay loam soil (Sharifi et al., 2011). The N_0 is partially derived from labile organic N and can be operationally defined as distinct pools of mineralizable N (Sharifi et al., 2008; Nyiraneza et al., 2012; St. Luce et al., 2013). These include Pool I, the N mineralized from the labile organic N pool from 0 to 2 wk; Pool II, the N mineralized from labile organic N during an intermediate time period (e.g., between 2 and >24 wk); and Pool III, a stable organic N pool that could potentially mineralize during soil incubation. Pool I typically increases with 10 to 15 yr of manuring (Sharifi et al., 2008, 2011), regardless of soil texture, whereas, Pool II and Pool III are less responsive to manure-amendment history in fine- than coarse-textured soils (Sharifi et al., 2011). The positive response of Pool I to a 10-yr manuring history led to the development of indices that considered Pool I alone or Pool I plus the initial soil mineral N concentration to represent plant-available N (Sharifi et al., 2011, 2014). Whether these indices are appropriate for predicting mineralizable N parameters in soils with a 3-yr history of manure application is not known.

Lengthy soil incubations are needed to quantify the N_0 and k, as well as biologically active pools of mineralizable N (Pools I and II). It would be preferable to find indicators to more rapidly assess mineralizable N in manure-amended soils. For instance, extractable organic C and N fractions that are strongly correlated to Pool I or Pool II could serve as rapid indicators of the mineralizable N pool because they can be quantified in a matter of days, rather than weeks (Pool I) or months (Pool II). It could also imply that those extractable organic C and N fractions are involved in the N mineralization process. Chemically extractable mineral N pools and organic C and N fractions hold promise in this regard, since they are bioavailable to the microorganisms that mediate the N mineralization process (Haynes, 2005) and are predictors of mineralizable N parameters (Dessureault-Rompré et al., 2010; Ros et al., 2011b; Haney et al., 2012; St. Luce et al., 2013, 2014). Nitrogen mineralization is related to the water and salt-extractable C and N concentrations (Murphy et al., 2000; Curtin et al., 2006; Haney et al., 2012), particulate organic matter (Willson et al., 2001; Sharifi et al., 2008; St. Luce et al., 2013, 2014) and microbial biomass (Carter and MacLeod, 1987; Deng et al., 2000; St. Luce et al., 2014) in a variety of soil textures under different fertilization regimes. It was hypothesized that one or more of the chemical extractions for mineral N and organic matter fractions are related to mineralizable N parameters in contrasting soil textures with a 3-yr manuring history.

The first objective of this study was to determine the mineralizable N parameters (e.g., $N_{0,}$ k, Pool I, Pool II, Pool III) of two soils with contrasting textures and a 3-yr history of LSM, LCM, and SPM manure application. The second objective was to evaluate the relationships between the biologically active N pools and chemically extractable forms of organic C, organic N, and mineral N, with particular emphasis on robust indicators of total N mineralized (TNM) that responded consistently in the contrasting soil textures.

MATERIALS AND METHODS

Field Site

The experimental sites were located at the Laval University Experimental Farm in Saint-Augustin-de-Desmaures, near Quebec City, Canada (46°44′ N, 71°31′ W; altitude, 110 m) on a poorly drained silty clay (432 g clay kg^{-1}, 163 g sand kg^{-1}, 35 g organic C kg^{-1}, 2.7 g total N kg^{-1}, pH = 7.0) classified as a mixed frigid dystric eutrudept (soil series: Tilly) and a well-drained sandy loam (170 g clay kg^{-1}, 680 g sand kg^{-1}, 19 g organic C kg^{-1}, 1.5 g total N kg^{-1}, pH = 6.8) classified as a mixed frigid typic dystrudept (soil series: Batiscan). In 2008, both sites were cropped with silage corn (*Zea mays* L.) and the residue was removed, leaving stubble <15 cm. Experimental plots (5 × 7 m) on both soils were fertilized in May 2009 with LSM, LCM, and SPM, as well as NPK and CTL. Manure and NPK treatments were applied by surface broadcasting and incorporated by harrowing to a depth of 0.1 m on the day of application to minimize ammonia volatilization. The CTL plots were also harrowed at this time. The three replicate plots of each treatment, arranged in a randomized complete block design, were fertilized the same way in 2009, 2010, and 2011, before planting soybean (*Glycine max*), wheat (*Tritium aestivum*), and corn, respectively. Aboveground crop residues were removed by hand in the fall after harvest each year, leaving <15 cm of aboveground stubble.

The manure amendments applied in this study were collected each year immediately before field application. For each year, the LSM was obtained from a commercial farrow-to-finish operation and was collected after about 6 mo of storage. The LCM was obtained after about 6 mo of storage from the Deschambault Research Center (Research and Development Institute for the Agri-Environment). Fresh SPM was obtained from a commercial broiler operation and included bedding material composed of wood shavings. The SPM was covered and stored for approximately 1 mo before use. Each year, fertilized plots received a target rate of 90 kg available N ha^{-1} (112.5 kg total N ha^{-1} for manure-amended soils) based on local recommendations (CRAAQ, 2010). For the manure amendments 70% of LCM and SPM, and 90% of LSM total N is considered available to cereals when applied in early spring and rapidly incorporated into the soil (CRAAQ, 2010). To simplify field work, an average coefficient of 0.8 was used to calculate field application rates for LCM, SPM, and LSM. This coefficient accounts for manure N losses due to ammonia volatilization and the availability of manure organic N. The cumulative total N added by the manure applications over 3-yr was 337.5 kg N ha^{-1}. Mineral N fertilizer was applied as 90 kg N ha^{-1} from calcium-ammonium-nitrate (Ca-NH_4NO_3), with triple superphosphate (8.7 kg P ha^{-1} for the sandy loam; 13.1 kg P ha^{-1} for the silty clay), and potassium chloride (16.6 kg K ha^{-1} for both soils) added based on provincial soil recommendations for Quebec, Canada (CRAAQ, 2010). No treatments were applied in 2012.

Manure Analysis and Soil Sampling

On each manure application date in 2009, 2010, and 2011, for each soil texture, a composite sample of each manure type was collected and analyzed (Table 1). The LCM and LSM were homogenized using a Polytron (Model PT 3100; Kinematica AG, Littau-Lucerne, Switzerland). Distilled water was added to the SPM (10:1, water/SPM ratio) and homogenized with the Polytron to make a slurry solution. The pH of LCM, LSM, and SPM was measured by direct reading with a glass electrode. Dry matter of the manure was determined as the mass of materials remaining after drying 100 mL of LCM and LSM and 50 g of fresh SPM for 96 h at 55°C. Total C was measured by dry combustion for SPM or by injecting 50 μL of homogenized LSM and LCM, into an automated combustion C analyzer (Model Formacs; Skalar Analytical, De Breda, The Netherlands). Total N and P concentrations were determined by measuring NH_4–N and PO_4–P concentrations in Kjeldahl acid digests with an automated colorimeter (QuickChem 8000 FIA+; Lachat Instruments, Loveland, CO). The NH_4–N and NO_3–N content of the liquid manures or SPM slurry were determined by shaking 10 mL of the sample with 50 mL of 1 mol L^{-1} KCl for 60 min. The NH_4–N and NO_3–N [NO_3–N + nitrite (NO_2–N)] concentrations in the extracts were measured using the colorimeter as described above. The NO_3–N concentrations were not provided as they were <0.005 g L^{-1} for the liquid manures and <0.016 g kg^{-1} for SPM.

Eight soil samples (0- to 20-cm depth) were collected with a soil auger (10-cm diam.) from each plot in spring 2012, composited and passed through a 2-mm sieve while field-moist. The soil was immediately refrigerated (4°C) for 2 wk until it was removed and for the 48 wk aerobic incubation or analyzed according to the standard procedure for each chemical extraction method (either on field-moist or air-dried soil).

Table 1. Physical and chemical characteristics (wet volume or mass basis) of the animal manures applied in 2009, 2010, and 2011 to sandy loam and silty clay soils.

Manure†	DM‡	C total	N total	NH_4–N	P total	pH
			2009			
LSM, g L^{-1}	38.7 (4.43)§	16.8 (1.80)	5.1 (0.35)	2.7 (0.12)	0.8 (0.09)	7.3 (0.21)
LCM, g L^{-1}	47.3 (4.03)	22.2 (1.48)	2.4 (0.06)	1.6 (1.20)	0.5 (0.01)	6.5 (0.07)
SPM, g kg^{-1}	661.1 (29.42)	271.1 (13.93)	23.3 (1.54)	5.1 (0.03)	9.8 (0.54)	8.8 (0.14)
			2010			
LSM, g L^{-1}	114.6 (6.59)	49.9 (4.29)	8.4 (0.13)	5.4 (0.71)	2.0 (0.01)	7.3 (0)
LCM, g L^{-1}	47.0 (2.48)	21.5 (1.36)	2.5 (0.04)	1.3 (0.03)	0.4 (0.02)	6.5 (0.14)
SPM, g kg^{-1}	769.1 (18.0)	331.1 (10.48)	30.3 (2.33)	3.68 (0)	9.8 (0.97)	8.8 (0.14)
			2011			
LSM, g L^{-1}	62.5 (2.97)	24.5 (1.12)	8.9 (0.06)	6.0 (0.03)	1.4 (0.02)	7.1 (0.02)
LCM, g L^{-1}	68.7 (4.60)	28.3 (1.17)	3.3 (0.08)	1.3 (0.15)	0.6 (0.07)	6.9 (0.07)
SPM, g kg^{-1}	796.5 (34.6)	323.3 (11.8)	29.7 (1.32)	4.6 (0.29)	10.7 (0.38)	8.4 (0.38)

† LSM, liquid swine manure; LCM, liquid dairy cattle manure; SPM, solid poultry manure.

‡ DM, dry matter.

§ Numbers in parenthesis following mean values represent SD.

Long-Term Aerobic Incubation

The 48-wk aerobic incubation was based on Sharifi et al. (2011) with minor modifications. Thirty grams of air-dry sandy loam soil was mixed with 30 g of acid-washed Ottawa sand (Thermo Fisher Scientific, Waltham, MA). The silty clay soil was mixed with 60 g of Ottawa sand due to its finer texture. The sand/soil mixes were added to Buchner funnel cylinders (Ellert and Bettany, 1988; Benedetti and Sebastiani, 1996) with Whatman GF/A 55 mm diameter filters lining the bottom. The water-filled pore space (WFPS) was maintained at 55% and the temperature set at 25°C in a dark controlled environmental chamber for 48 wk. The soils were leached by vacuum suction (ME 16C NT, VACUUBRAND INC., Essex, CT) with 0.01 mol L^{-1} $CaCl_2$ followed by a zero-N nutrient solution (0.002 mol L^{-1} $CaSO_4$, 0.002 mol L^{-1} $MgSO_4$, 0.005 mol L^{-1} $Ca(H_2PO_4)_2$, and 0.0025 mol L^{-1} K_2SO_4) every 2 wk for the first 12 wk and every 4 wk thereafter (Curtin and Campbell, 2008). Leachates were analyzed colorimetrically for NH_4–N and NO_3–N using a modified indophenol blue technique (Sims et al., 1995).

Potentially mineralizable N was calculated using a first-order, nonlinear kinetic model using the Marquardt iteration method (Sharifi et al., 2011; St. Luce et al., 2013):

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

where, $N_{\min}$ is the cumulative N mineralized (NH_4–N + NO_3–N) in time t (Pool II), N_0 is potentially mineralizable N, and k is the mineralization rate constant. The mineralized N was separated into two distinct pools and a third pool was calculated. Pool I was measured at 2-wk following rewetting and was not included in the calculation of N_0. Pool II was the cumulative N mineralized between 2 and 48 wk. Pool III was calculated as the difference between the predicted N_0 and Pool II (Sharifi et al., 2007). Calculation of N_0 mean values using first-order kinetics was not possible for the silty clay soil as the majority of soils showed a linear increase in mineral N over 48 wk of incubation and did not converge in a nonlinear, first-order kinetic model. A simple linear (zero-order) regression model based on Griffin and Honeycutt (2000) and Griffin et al.(2005) was also used to describe the cumulative N mineralization between 2 and 48 wk because of the linear relationship between cumulative mineralization and time in both soil textures:

$$N_{\min} = b(t) + a \qquad [2]$$

where, a is the Y-axis intercept, b is the slope, and t is time (wk).

Total N mineralized (TNM) was calculated as the cumulative N mineralized after the first leaching event and was not corrected by subtracting the CTL from the manure-amended soils:

$$\text{TNM} = \text{Pool I} + \text{Pool II} \qquad [3]$$

Extractable Organic C and N Fractions

Water and hot-water extractions were conducted sequentially based on Curtin et al. (2006) and Chantigny et al. (2010). In short, 5 g of air-dried soil was extracted with 30 mL of 20°C deionized water for determination of water extractable organic C and N (WEOC and WEON). Promptly, 30 mL of 20°C deionized water was then added to the remaining soil and mixed thoroughly, and placed in a 50°C water bath for 16 h for determination of hot-water extractable organic C and N (hot-WEOC and hot-WEON). The WEON and hot-WEON concentrations were the difference between total N concentration after persulfate oxidation and the mineral N concentration of non-oxidized samples, respectively (Cabrera and Beare, 1993). Total and mineral N concentrations of oxidized and non-oxidized samples were determined colorimetrically using a modified indophenol blue technique (Sims et al., 1995). The organic C concentrations in WEOC and hot-WEOC extracts were measured with a Sievers Innovox TOC analyzer (GE Analytical Instruments, Boulder, CO).

The microbial biomass C and N (MBC and MBN) analysis was done by chloroform fumigation-direct extraction with 10 g of field-moist soil and extracted with 0.5 mol L^{-1} K_2SO_4 (Voroney et al., 2008). The MBC was calculated as total organic C in fumigated samples minus the total organic C in non-fumigated samples, both analyzed with a Sievers Innovox TOC analyzer (GE Analytical Instruments, Boulder, CO). The total C in MBC was divided by 0.45, a coefficient of extraction efficiency for MBC (Wu et al., 1990; Joergensen, 1996). The MBN was calculated as total N concentration in fumigated sample minus the total N concentration in non-fumigated sample, based on the NO_3–N concentration of persulfate oxidized samples (Cabrera and Beare, 1993) measured colorimetrically using the modified indophenol blue technique (Sims et al., 1995). The total N in MBN was then divided by a correction factor of 0.54 (Joergensen and Mueller, 1996), a coefficient of extraction efficiency for MBN. The total organic C and total N minus the mineral N concentration in the non-fumigated samples were considered salt extractable organic C and N (SEOC and SEON), respectively (Chantigny et al., 2008).

For particulate organic matter C and N (POMC and POMN), 25 g of air-dried soil was dispersed in 100 mL of a 5 g L^{-1} sodium hexametaphosphate solution in a 250-mL nalgene bottle by shaking for 16 h, and then dispersed soil was passed through a 53-µm sieve (Gregorich and Beare, 2008). The retained sand and macroorganic matter were air-dried overnight and then oven-dried at 50°C for 24 h. The concentration of POMC, POMN, total C, and total N of each soil was determined by dry combustion with a Flash EA 1112 series CN analyzer (ThermoFinnigan, Italy) after passing through a 250-µm sieve.

Extractable Mineral Nitrogen Pools

For KCl-exchangeable NH_4–N and NO_3–N (KCl-NH_4 and KCl-NO_3) concentrations, 5 g of field-moist soil was extracted with 50 mL of 2 mol L^{-1} KCl and their sum defined as KCl-Total. The $CaCl_2$–exchangeable NH_4–N and NO_3–N ($CaCl_2$–NH_4 and $CaCl_2$–NO_3) were removed by leaching at $t = 0$ of the long-term aerobic incubation and their sum defined as $CaCl_2$–Total. The water extractable mineral N (WEMN) and hot-water extractable mineral N (hot-WEMN) were the mineral N concentrations in the non-oxidized WEON and hot-WEON, respectively. Immediately following the hot-WEON extraction and filtration, 30 mL of 2 mol L^{-1} KCl was added to each soil to determine post hot-WEON-NH_4, which represents exchangeable NH_4–N released during the hot-WEON extraction process. The samples were measured for NH_4–N and/or NO_3–N using a modified indophenol blue technique (Sims et al., 1995).

Statistical Analysis

Data were analyzed with SAS 9.3 software (SAS Institute Inc., 2011). Data was first tested for normality using Shapiro-Wilks W test. The effects of amendment on mineralizable N parameters were analyzed by one-way analysis of variance (ANOVA) for each soil texture separately using the PROC MIXED statement with block as a random factor. When the amendment effect was significant, differences between treatment means were evaluated using the Tukey Honestly-Significant-Difference post-hoc test. Pre-planned orthogonal contrast analysis was then used to test for differences between (i) the CTL and N fertilized soils (NPK, LSM, LCM, and SPM), (ii) the NPK and manure-amended soils, and (iii) the liquid manure-amended soils (LSM vs. LCM). Pearson correlation coefficients (r) were calculated using the PROC CORR statement to determine the strength of relationships between labile organic C and N fractions, C/N ratios, and mineral N pools with Pool I, Pool II, and TNM with replicate data. Simple linear regressions between the cumulative N mineralized and time (2–48 wk) were calculated using the PROC REG statement with replicate data.

RESULTS AND DISCUSSION

Mineralizable Nitrogen in Soils with Three-Year Manuring History

Pool I was the only mineralizable N pool to be significantly affected by amendment in the sandy loam and silty clay soil ($P < 0.05$ and 0.001, respectively; Table 2). The average value for Pool I in manure-amended soils was 16 mg N kg^{-1} in the sandy loam and 12 mg N kg^{-1} in the silty clay, which is within the Pool I range of 4 to 61 mg N kg^{-1} of other arable soils in humid temperate regions of Canada (Sharifi et al., 2007, 2008; Dessureault-Rompré et al., 2010; Nyiraneza et al., 2012; St. Luce et al., 2013).

Pre-planned orthogonal contrasts for Pool I were significant in the sandy loam soil. Pool I was higher in the CTL than the N fertilized (NPK, LSM, LCM, SPM) soils (26 vs. 17 mg N kg^{-1}) and it was higher in NPK than the manure-amended soils (19 vs. 16 mg N kg^{-1}). The LSM had more Pool I than LCM (23 vs. 14 mg N kg^{-1}) in the sandy loam soil. In the silty clay soil, pre-planned contrast analysis indicated higher Pool I concentration with LCM than LSM (18 vs. 11 mg N kg^{-1}). On average, the values of Pool I represent a potential in-field N supply of 45 and 29 kg N ha^{-1} in the sandy loam and silty clay manure-amended soils, respectively. This translates into about 38 to 50% and 24 to 32% of wheat N uptake in the sandy loam and silty clay soil, respectively, based on recommendations of 90 to 120 kg N ha^{-1} (CRAAQ, 2010). For grain corn, Pool I represented about 26 to 38% and 17 to 24% of the 120 to 170 kg N ha^{-1} requirement in this region in the sandy loam and silty clay soils, respectively (CRAAQ, 2010).

Pool I was two-fold lower in SPM than the CTL in the sandy loam soil. The lower Pool I concentration in the SPM sandy loam soil could indicate that mineralizable N was lost (e.g., through plant uptake or other loss pathways) since the SPM application nearly 1 yr earlier or it may be a result of undecomposed wood chip bedding releasing labile C and immobilizing N during the first 2 wk of aerobic incubation. The latter possibility is consistent with in-field data from 2010 and 2011 that showed springtime CO_2 emissions increased 10 to 45% following application of SPM compared with other manure types (Pelster et al., 2012), which implies rapid microbial growth and N immobilization.

In the silty clay soil, Pool I was significantly greater in LCM than other treatments. The organic N in cattle manure is more stable than swine and poultry manure (Chadwick et al., 2000) and could be physically protected in this fine-textured soil (Serna and Pomares, 1992). Conservation of readily mineralizable N (Pool I) through physicochemical mechanisms also explains why LCM-amended soils retained more mineralizable N in the silty clay than the sandy loam soils. The hypothesized organic N conservation mechanism is an interaction between soil texture (substrate accessibility) and the decomposability of the manure (substrate). This mechanism of physical protection of readily mineralizable N is supported by Rochette et al. (2006) who found in situ manure C decomposition rates of solid and liquid dairy manures were similar in a loamy soil, but the solid manure (less decomposable substrate) decomposed less than the liquid manure in the clay soil. This suggests that the relationship between a substrates decay and sand or clay content is nonlinear, and strongly dependent on the substrates stability. Soil amendments decay disproportionally slower in fine- than coarse-textured soils. This resulted in more carryover of mineralizable N in the sandy loam than silty clay soil and supports the concept that chemicals exhibit different residence times based on their protection from decay (Schmidt et al., 2011). Furthermore, it also suggests that decay series, or coefficients of N availability need to be formulated based on manure type and soils texture versus manure type alone as is currently practiced in Quebec, Canada (CRAAQ, 2010). Our data, N_2O emission data (Pelster et al., 2012) and work in Quebec, Canada by Rochette et al. (2006) suggests that N dynamics depend on an interaction between manure type and

Table 2. Effect of 3-yr amendment history on potentially mineralizable N (N_0), mineralization rate coefficients (k), three pools of mineralizable N (Pool I, cumulative amount of N mineralized in the first 2-wk following rewetting; Pool II, cumulative amount of N mineralized between 2 and 44 wk; Pool III, N_0 minus Pool II) and Total N mineralization (TNM, Pool I + Pool II).

Treatment† (n = 3)	Mineralizable N parameters					
	N_0	k	Pool I	Pool II	Pool III	TNM
Sandy loam	mg N kg^{-1}	wk^{-1}	———— mg N kg^{-1} ————			
CTL	155 (43)‡	0.019 (0.003) a§	26 (4) a	85 (14)	70 (29)	110 (17)
NPK	155 (28)	0.018 (0.002) a	19 (4) ab	84 (14)	71 (21)	103 (11)
LSM	184 (21)	0.013 (0.0004) ab	23 (4) ab	85 (8)	99 (13)	108 (12)
LCM	229 (84)	0.010 (0.003) b	14 (8) ab	81 (13)	160 (85)	95 (21)
SPM	246 (5)	0.009 (0.0004) b	12 (5) b	80 (4)	166 (4)	91 (9)
Silty clay						
CTL	ND¶	ND	10 (0.9) b	63 (13)	ND	72 (13)
NPK	ND	ND	9 (3) b	70 (5)	ND	79 (8)
LSM	ND	ND	11 (1) b	72 (1)	ND	83 (0.3)
LCM	ND	ND	18 (0.7) a	77 (10)	ND	95 (11)
SPM	ND	ND	6 (0.5) b	63 (9)	ND	69 (9)
			ANOVA (*P-values*)			
Source of variation						
Sandy loam						
Treatment	0.11	0.014	0.017	0.84	0.09	0.17
Silty clay						
Treatment	ND	ND	0.0005	0.88	ND	0.45
			Contrasts# (*P-values*)			
Sandy loam						
CTL vs. Fertilizer	NS	0.012	0.013	0.71	0.087	0.13
NPK vs. Manure	NS	0.030	0.041	0.85	0.18	0.26
LSM vs. LCM	NS	0.13	0.030	0.56	0.10	0.14
Silty clay						
CTL vs. Fertilizer	ND	ND	0.40	0.47	ND	0.44
NPK vs. Manure	ND	ND	0.056	0.36	ND	0.29
LSM vs. LCM	ND	ND	0.0013	0.73	ND	0.41

† CTL, zero-N control; NPK, N, P, and K fertilizer; LSM, liquid swine manure; LCM, liquid dairy cattle manure; SPM, solid poultry manure; NS, non-significant.

‡ Numbers in parenthesis following treatment means represent SE.

§ Means in the same column with the same letter were not significantly different ($P < 0.05$, post-hoc Tukey HSD test).

¶ ND, not determined because data did not fit the non-linear, first-order kinetic model.

\# CTL vs. N Fertilizer, (CTL vs. NPK, LSM, LCM, and SPM); NPK vs. Manure, (NPK vs. LSM, LCM, and SPM).

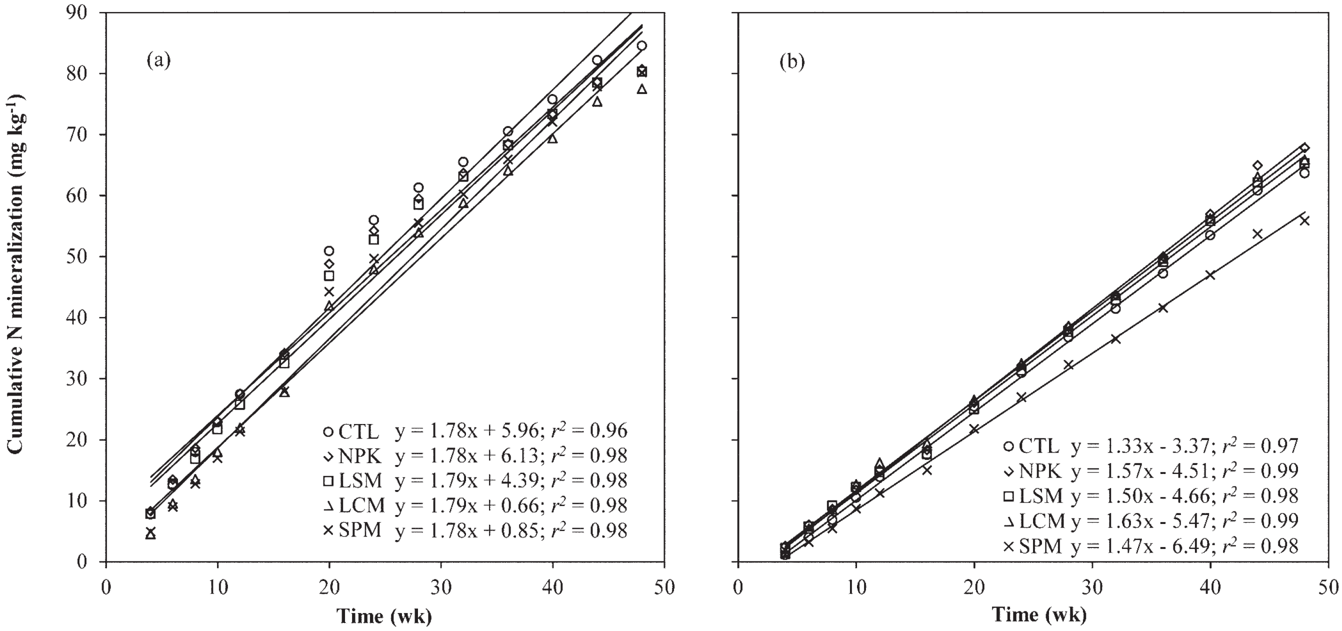

Fig. 1. Cumulative N mineralized between 2 and 48 wk in an aerobic incubation (Pool II) of (a) sandy loam and (b) silty clay soils. These soils had a 3-yr history of annual applications of N, P, and K fertilizer (NPK), liquid swine manure (LSM), liquid dairy cattle manure (LCM) and solid poultry manure (SPM) applied at 90 kg available N ha^{-1} yr^{-1}, and a zero-N control (CTL). Data points were mean values (*n* = 3) and standard error of the mean (not shown for visual clarity) did not exceed 21%.

soil texture. Future work should determine whether in situ residual N availability coefficients require modification based on interactions between manure type and soil texture to maximize the N fertilizer value of manure, and reduce the risk of N loss to the environment.

Pool II was not significantly affected by amendment in the sandy loam or silty clay soil (Table 2) and average values were 82 mg N kg^{-1} in sandy loam and 71 mg N kg^{-1} in silty clay soils with 3-yr manuring history. This is within the range of 10 to 138 mg N kg^{-1} reported in arable soils from temperate humid regions of Canada (Sharifi et al., 2007, 2008; Dessureault Rompré et al., 2010; Nyiraneza et al., 2012; St. Luce et al., 2013). Cumulative N mineralized (Pool II) was linear over 48 wk (r^2 = 0.96–0.99; Fig. 1) and the average slope in the sandy loam and silty clay soils with a history of manure-amendment was 1.79 and 1.53 mg N kg^{-1} wk^{-1}, respectively. This is consistent with previous findings of steeper slopes (greater N mineralization rate) in sandy than clay soils (Ros et al., 2011b).

Pool III and N_0 were not affected by amendment in the sandy loam and could not be determined in the silty clay (Table 2). The average value for Pool III was 142 mg N kg^{-1} in sandy loam soils with a 3-yr manuring history and was within the range of 0 to 206 mg N kg^{-1} reported in a clay loam soil with a long-term history of fresh or composted beef cattle manure containing straw or woodchip bedding in southern Alberta, Canada (Sharifi et al., 2014). Pool III in the CTL and NPK (70 and 71 mg N kg^{-1}, respectively) were near the maximum range of 1 to 78 mg N kg^{-1} in arable soils from humid temperate regions of Canada (Sharifi et al., 2007, 2008; Dessureault-Rompré et al., 2010; Nyiraneza et al., 2012). The average N_0 for manure-amended sandy loam soils (220 mg N kg^{-1}) was within the range of the 18 to 277 mg N kg^{-1} reported for arable soils in humid temperate regions of Canada (Sharifi et al., 2007; Dessureault-Rompré et al., 2010; Sharifi et al., 2011; Nyiraneza et al., 2012; St. Luce et al., 2013).

There was a two-fold higher *k* in the NPK and CTL treatments than SPM and LCM treatments in the sandy loam soil (Table 2). However, the average *k* of 0.01 wk $^{-1}$ in sandy loam soils with a history of manure-amendment was lower than the 0.056 wk^{-1} reported in a medium-textured sandy clay soil with a 10-yr history of semi-solid cattle manure in New Brunswick, Canada (Sharifi et al., 2011). Contrast analysis of sandy loam soils indicated that *k* was significantly higher in the CTL than the N fertilized soils (0.019 vs. 0.013 mg N kg^{-1}) and in the NPK than the manure-amended soils (0.018 vs. 0.011 mg N kg^{-1}). Sharifi et al. (2011) showed that *k* was not influenced by a 10-yr history of manure in sandy loam and sandy clay soils compared with unmanured soils. On average, the higher *k* values in the unmanured versus manured soils implies that the half-life of N_0 depletion, or formation will be shorter in the unmanured (CTL and NPK) than manured soils based on the first-order kinetic model. This suggests that the stability of organic N is higher in the manured soils based on the parameters (N_0 and *k*) estimated by the first-order kinetic model because at any point in time, the N mineralization rate is a product of *k* and the N_{min} (cumulative N mineralized).

Indicators of Nitrogen Mineralization Parameters

Biological Parameters

Pool I was significantly correlated to Pool II (*r* = 0.78) in the sandy loam soil but not in the silty clay soil. When Pool I and post-hot-WEON-NH_4 were combined, it improved the correlation with Pool II (*r* = 0.87) and with TNM (*r* = 0.96) in the sandy loam soil. Pool I was more strongly correlated with TNM in the sandy loam soil (*r* = 0.92) than the silty clay soil (*r* = 0.64).

This is supported by a larger proportion of readily mineralizable N relative to TNM in the sandy loam soil than the silty clay soil. Even though Pool I was included in TNM, it represented only 19 and 14% of TNM over 48 wk in the sandy loam and the silty clay soil, respectively. Therefore, we consider Pool I a robust biological indicator of N mineralization (Fig. 2). Future work should verify that Pool I works as a robust indicator of in situ N mineralization for a wide range of soils in humid temperate regions. Furthermore, assessment of whether Pool I is a suitable indicator of N loss during the non-growing season warrants investigation in manure-amended soils of northern humid temperate regions because losses of organic N in the non-growing season have approached one-half (~20–32.5 kg N ha^{-1}) of residual manure organic N remaining at harvest (Chantigny et al., 2014). Implying that organic N mineralization is a dominant loss pathway during the non-growing season. Currently, the soil mineral N at harvest is used to estimate non-growing season losses of N (De Jong et al., 2007) but such indicators ignore the potential contribution of organic N to these losses. The use of an indicator for mineralizable N that is lost during the non-growing season in manure-amended soils may be required in the next generation of indicators for N loss risk (Chantigny et al., 2014).

Chemical Parameters

Organic Nitrogen Fractions. The SEON was the only chemical index significantly correlated to a mineralizable N pool, that is, Pool I, in the silty clay (r = 0.74) and sandy loam soil (r = 0.52; Table 3). Neutral and acidic salt solutions extract a biologically active N fraction (Ros et al., 2011a), and Pool I is considered the most biologically active N fraction in aerobic incubations, representing labile organic N with the quickest turnover time (Sharifi et al., 2011). In the sandy loam soil, POMN was significantly correlated to Pool I (r = 0.55), while SEON, POMN, and Total N were significantly correlated to Pool II and TNM (r = 0.65–0.75; Table 3). Grouping the sandy loam and silty clay soils together weakened the correlation coefficients and often led to two clusters of data with trend lines of different slopes (data not shown) between chemical indices and mineralizable N. This supports previous research in soils with a 10-yr history of manure-amendment (Sharifi et al., 2011), arable soils with a broad range of management history (Serna and Pomares, 1992; Nyiraneza et al., 2012) and grassland soils with a range of physicochemical properties (McDonald et al., 2014); that found stronger relationships between N mineralization parameters and chemical indices of N availability when grouping soils based on texture.

Organic Carbon Fractions. Total C, SEOC, and POMC were significantly correlated with Pool I (r = 0.74, 0.72, and 0.66, respectively) in the sandy loam soil, but not in the silty clay soil (Table 3). Total C was most strongly correlated to Pool II in the sandy loam soil (r = 0.89) followed by WEOC, SEOC, and POMC (r = 0.67–0.70). The hot-WEOC was the only organic C fraction to be significantly correlated to Pool II in the silty clay soil (r = 0.58). Total C was more strongly correlated to TNM in the sandy loam soil (r = 0.88) followed by WEOC, SEOC, and POMC (r = 0.70–0.75). This is consistent with Ros et al. (2011b), who reported that mineralizable N was most strongly related to C, in particular, the soluble C in hot-water extracts. The strong positive relationship between C and mineralizable N provides evidence that C availability limited N mineralization in the sandy loam soil but not the silty clay soil. This is supported by the background total C concentration being almost two-fold lower in the sandy loam than silty clay soil, which implies applied C from manure has a more important role controlling N mineralization in coarse- than fine-textured soils.

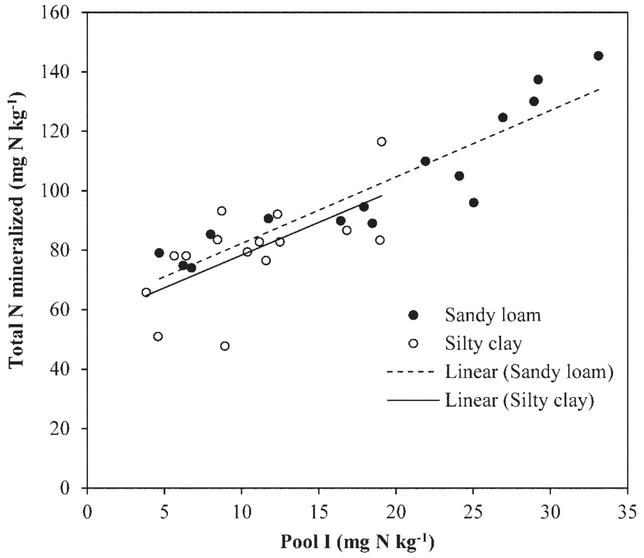

Fig. 2. The functional relationship between total N mineralized (Pool I + Pool II) and Pool I during a 48-wk aerobic incubation of sandy loam and silty clay soils. These soils had a 3-yr history of annual applications of N, P and K fertilizer (NPK), liquid swine manure (LSM), liquid dairy cattle manure (LCM) and solid poultry manure (SPM) applied at 90 kg available N ha^{-1} yr^{-1}, and a zero-N control (CTL).

Carbon/Nitrogen Ratios. The WEOC/WEON ratio was the only C/N ratio to be significantly correlated to a mineralizable N (Pool I) in the sandy loam soil (r = 0.52; Table 3), while POMC/POMN was the only C/N ratio to be significantly correlated to a mineralizable N pool (Pool II) in the silty clay soil (r = 0.57). This may indicate that the concentration of water soluble C relative to N is more related to the mineralization of the labile organic N with the fastest turnover time (Sharifi et al., 2011), while in the silty clay soil the POMC/POMN is more related to the mineralization of the intermediate pool of organic N, namely the partially decomposed POM. This ultimately infers that the C/N ratios of labile organic matter have different meanings in these contrasting soil textures. Overall, the lack of significant inverse correlations between C/N ratios and N mineralization parameters implies that N did not limit the microbial assimilation of C during the incubation, which is supported in the sandy loam soil by the positive relationship of N mineralization parameters Pool I, Pool II, and TNM, with Total C, SEOC, and POMC.

Mineral Nitrogen Pools. The WEMN and hot-WEMN were most strongly correlated to Pool I in the sandy loam soil (r = 0.68 and 0.67, respectively; Table 3). Post-hot-WEON-NH_4 was

Table 3. Soil chemical and biological parameters correlated with mineralized N (Pool I, Pool II and TNM, total N mineralized) in sandy loam and silty clay soils with a 3-yr amendment history. Values are Pearson correlation coefficients calculated from 15 observations for each soil type.†

	Correlation coefficient					
	Sandy loam			Silty clay		
Parameter	Pool I	Pool II	TNM	Pool I	Pool II	TNM
Pool I						
Pool I		0.78	0.92		0.41	0.64
Pool I + Post hot-WEON-NH_4		0.87	0.96		0.28	0.48
Organic N						
WEON	-0.32	-0.07	-0.18	0.10	-0.30	-0.22
hot-WEON	-0.05	-0.28	-0.20	0.40	0.24	0.32
MBN	0.10	0.10	0.11	-0.06	-0.33	-0.30
SEON	0.52	0.75	0.70	0.74	0.32	0.48
POMN	0.55	0.74	0.70	0.12	0.15	0.16
Total N	0.49	0.70	0.65	0.44	0.08	0.20
Organic C						
WEOC	0.61	0.69	0.70	0.00	0.24	0.20
hot-WEOC	0.08	0.32	0.24	0.02	0.58	0.49
MBC	0.29	0.37	0.35	-0.12	-0.35	-0.33
SEOC	0.72	0.70	0.75	0.49	0.06	0.19
POMC	0.66	0.67	0.70	0.13	0.34	0.32
Total C	0.74	0.89	0.88	0.10	0.37	0.35
C/N ratios						
WEOC/WEON	0.52	0.39	0.46	-0.11	0.29	0.21
hot-WEOC/hot-WEON	0.22	0.47	0.39	-0.34	0.32	0.17
MBC/MBN	-0.03	-0.13	-0.09	-0.09	0.04	0.01
SEOC/SEON	0.14	-0.11	-0.01	-0.32	-0.30	-0.35
POMC/POMN	-0.28	-0.34	-0.33	-0.02	0.57	0.48
Total C/N	-0.13	-0.27	-0.23	-0.43	0.17	0.01
Mineral N						
KCl-NO_3	0.59	0.66	0.66	0.18	0.31	0.31
KCl-NH_4	-0.30	-0.19	-0.25	0.12	0.18	0.18
KCl-Total	0.39	0.49	0.48	0.22	0.36	0.37
$CaCl_2$–NO_3	0.56	0.59	0.61	0.46	0.37	0.45
$CaCl_2$–NH_4	0.36	0.18	0.27	-0.26	-0.29	-0.32
$CaCl_2$–Total	0.62	0.62	0.66	0.45	0.37	0.44
WEMN	0.68	0.61	0.67	0.01	0.16	0.14
hot-WEMN	0.67	0.62	0.68	-0.30	-0.09	-0.16
Post hot-WEON-NH_4	0.56	0.88	0.80	-0.12	-0.17	-0.18

† $r < 0.52$ is not significant at the 0.05 probability level; $r \geq 0.52$ is significant at the 0.05 probability level; $r \geq 0.66$ is significant at the 0.01 probability level; $r > 0.77$ is significant at the 0.001 probability level; No inverse relationships were significant.

most strongly correlated to Pool II and TNM in the sandy loam soil (r = 0.88 and 0.8). Post hot-WEON-NH_4 is sequentially extracted following removal of WEON and hot-WEON and is considered to be the exchangeable NH_4–N weakly adsorbed to clay minerals that is released by hydrolysis of organic N during the hot water extraction process (Curtin et al., 2006; Chantigny et al., 2010). The weak correlations between post hot-WEON-NH_4 and Pool II or TNM in the silty clay, but the strong correlations in the sandy loam implies that this extraction was more efficient removing NH_4–N associated with clays in the sandy loam soil. It may also extract NH_4–N by lysing cells of soil biota and plant residue fragments. Further evaluation of post hot-WEON-NH_4 is warranted, particularly in coarse-textured soils.

Post hot-WEON-NH_4 is sequentially extracted following removal of WEON and hot-WEON and is considered to be the exchangeable NH_4–N weakly adsorbed to clay minerals that is released during the 16 h, hot water extraction process (Chantigny et al., 2010). Sequentially, Pool II and post hot-WEON-NH_4 are related because they are measured after removal of the most labile organic N. Therefore, it is logical that Pool II and post hot-WEON-NH_4 would be strongly correlated. Although other researchers have investigated hot-KCl extractable NH_4–N (Gianello and Bremner, 1986; Sharifi et al., 2011; Nyiraneza et al., 2012), this fraction of N involves no prior sequential extractions to remove labile organic N and does not include organic N (Curtin et al., 2006). As post hot-WEON-NH_4 is an N mineralization process-based extraction it may represent a better indicator of a light-textured soils potential capacity to mineralize, especially the intermediate pool of mineralizable N–Pool II.

CONCLUSIONS

Pool I was the only mineralizable N pool significantly affected by manure type in both soil textures, the only mineralizable N pool to be significantly correlated to a chemical index (SEON) in each soil, and the only N availability index that was a consistent indicator of TNM for both soil textures. A 3-yr history of manure application can result in different quantities of residual readily mineralizable N (Pool I), but this appears to depend on an interaction between manure type and soil texture. The SPM application led to a reduction of Pool I in the sandy loam soil, and the LCM increased Pool I in the silty clay soil. This implies that residual N availability coefficients based on manure type and soil texture may require consideration to optimize supplemental N fertilizer recommendations and minimize N loss to the environment. Pool I was a sensitive measure and robust indicator of mineralizable N in both soil textures. We recommend 2-wk aerobic incubations for detecting readily mineralizable N in soils with a short-term history of manure amendment applied at recommended rates. The 2-wk incubations could be used for determining the residual manure N fertility value to optimize application rates of manure and supplemental N fertilizer.

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