COOL SEASON MINERALIZATION OF RECALCITRANT ORGANIC N IN UNDISTURBED CORES OF MANURED SOILS

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ABSTRACT

The objectives of this study were to (1) quantify cool season N mineralization from recalcitrant organic N in soils with a long history of manure application and (2) examine the relationship between cool season recalcitrant organic N mineralization and a fall mineralizable N index. "Cool season" was defined as October to February for fields with winter crops and October to May for fields with no winter crops. Two *in situ* trials were conducted with undisturbed soil cores in Oregon's Willamette Valley during the late fall, winter and early spring of 2003-2004 and 2004-2005. The ion exchange resin / soil core method (IER/SCM) was used.

On all soils tested, organic N mineralized during October to February, when crops would either not be present or would have very low uptake of mineral N. Four of the seven soils tested had net mineralization exceeding 20 ppm[1] N during this time period. Additional N mineralized between February and May. In the fall, most mineral N was present as NO_3^- in the soil mass. During the winter, most of the mineral N in each soil core leached to the resin below the soil. During the spring, NO_3^- levels increased in the soil and less leaching to the resin occurred. The recalcitrant organic N mineralization rate in the cool season was not correlated to the fall mineralizable N index.

INTRODUCTION

During the fall, winter and spring in temperate zone agriculture, precipitation often exceeds evapotranspiration and crop uptake of N is low. Because of this, residual soil NO_3^- (that which remains in the soil after summer crops are harvested) tends to leach or be lost via denitrification. NO_3^- leaching may contaminate groundwater; denitrification can generate the greenhouse gas N_2O. Some European states have set agricultural goals for residual soil NO_3^- to be below 40-62 lb N ac^{-1} (Hofman et al., 1994). In Washington, the USDA Nutrient Management standard specifies that residual soil NO_3^- in manured fields should be below 70 lb N ac^{-1} (USDA-NRCS, 2002; Sullivan and Cogger, 2003). Zebarth et al. (1995) estimated the acceptable upper limit of residual soil NO_3^- to be 89 lb N ac^{-1} in south-coastal British Columbia. These examples suggest 40-90 lb N ac^{-1} as a maximum acceptable concentration of residual soil NO_3^-. Assuming a sampling depth of 12 inches and bulk density of 1.4 g cm^{-3}, 40-90 lb N ac^{-1} equals 11-24 ppm N.

NO_3^- mineralized during the cool season is presumably as likely to leach or denitrify as residual soil NO_3^-. Cool season N mineralization in manured soils, however, has not been previously addressed in the literature. Cool season N mineralization is especially likely in fields with long histories of manure applications. This is because the pool of slowly mineralized, or

[1] Throughout this paper, ppm is used to represent parts per million (elemental N per dry soil mass) and is equivalent to mg N kg^{-1} soil.

recalcitrant, organic N compounds can grow until annual recalcitrant organic N mineralized approaches annual recalcitrant organic N applied (Fried et al., 1976). Recalcitrant organic N includes compounds associated with lignin from plant residues and animal waste, organic compounds and microbial matter adsorbed onto clay or contained inside soil aggregates, and humus (Van Veen et al., 1984).

MATERIALS AND METHODS

The experiments were conducted on soil from six farm fields (Table 4.1) in Oregon's Willamette Valley using an ion exchange resin/soil core method (IER/SCM) similar to that developed by DiStefano and Gholz (1986). The fields had not received manure, organic amendments or fertilizer within two months of core collection to ensure that little quickly mineralized (labile) organic N remained in the soil. Cores were constructed by driving 10 inch long aluminum pipes (4 inch diameter) to a depth of 8 inches and then using a shovel to excavate the pipes containing undisturbed soil. The cores were refrigerated (4°C) within five hours. Additional soil from each site was collected in bulk, sealed in clean plastic pails and refrigerated (4°C) within five hours. This bulk soil was used for baseline (pre-incubation) analysis of soil moisture and chemistry.

The bottom 1 inch of soil was scraped from each core and replaced with two ion exchange resin (IER) bags. The IER bags contained equal volumes of Sybron Ionac C-249 and ASB-1P. The upper bag (just below the soil inside the tube) contained 104 g moist IER and the lower bag (in contact with the soil outside of the tube) contained 80 g moist IER. The upper IER bag captured NO_3^- and NH_4^+ leaching out of the soil core. The lower IER bag prevented contamination of the upper bag from below. The cores were incubated in a randomized block in a field of soft white winter wheat (*Triticum aestivum* L.) near Forest Grove Oregon (45.59°N, 123.14°W), approximately 25 miles west of Portland. Incubation information is summarized in Table 4.2.

Oregon State University (OSU) recommends fertilizing pasture grasses with N at 200 growing degree days (GDD, base temperature = 0 °C) to coincide with accelerated crop growth at that time (Pirelli et al., 2004). In both years, cores were harvested in February at 200 GDD. Before this date, NO_3^- is especially vulnerable to leaching in fields with winter grass crops due to low crop N uptake. In 2004, a set of cores was also harvested in May because the period from 200 GDD to May poses NO_3^- leaching risks for fields that are fallowed during the winter and not planted to a crop until May.

Soils were analyzed for moisture content and a mineralizable N index (Horneck et al., 1989). A 20 g moist sub-sample was extracted with 100 ml 2M KCl (one hour on a reciprocating shaker) and filtered with a Whatman 934-AH glass microfibre filter or a Whatman Puradisc 25AS 0.45 um syringe filter. The filtered extracts were colorimetrically analyzed for NO_3^--N by cadmium reduction and diazotization and for NH_4^+-N by the alkaline phenol/hypochlorite method (Astoria-Pacific, 1998).

After incubation, the soil from each core was removed, weighed, mixed thoroughly and sub-sampled for moisture, NO_3^--N and NH_4^+-N analyses using the same methods used for baseline analyses. The lower IER bag was discarded. A 10 g moist sub-sample of the upper IER was extracted with four sequential 60 ml aliquots of 2M KCl with 15 minutes of shaking. The extract was analyzed for NO_3^--N and NH_4^+-N with the same colorimetric analyses used for the soils. This method of IER extraction yielded 95% of the NO_3^--N and 106% of the NH_4^+-N in a trial with standard solutions adsorbed onto IER.

Table 4.1. Field characteristics. Manure history "yes" indicates annual dairy manure applications for greater than 10 years; "no" indicates no manure applications in past 10 years.

Field	Soil	Classification	% Clay	%C	%N	C:N	Previous crop	Manure history
A	Newberg fine sandy loam	coarse-loamy, mixed, mesic fluventic haploxerolls	11.9	1.2	0.11	11.3	silage corn	Yes
B	McBee silty clay loam	fine-silty, mixed, mesic cumulic ultic haploxerolls	23.8	3.42	0.32	10.6	perennial grass	Yes
D	Woodburn silt loam	fine-silty, mixed, mesic aquultic argixerolls	13.1	3.94	0.32	12.3	silage corn	Yes
E	Wapato silty clay loam	fine-silty, mixed, mesic fluvaquentic haplaquolls	19.7	1.68	0.16	10.3	silage corn	Yes
F	McBee silty clay loam	fine-silty, mixed, mesic cumulic ultic haploxerolls	35	2.08	0.21	9.9	silage corn	No
G	Woodburn silt loam	fine-silty, mixed, mesic aquultic argixerolls	16.6	3.02	0.24	12.4	silage corn	Yes

% clay determined by pipette method, %C and %N determined by Leco CNS-2000 macro-analyzer, pH determined by 1:2 soil to water.
All analyses in this table were by the OSU Central Analytical Lab (Horneck et al., 1989).

Table 4.2. Incubation trials. Numbers in parentheses indicate the number of cores per field harvested on that date.

	2003-2004	2004-2005
Fields	A,D,E	A,B,F,G
Soil collection	3-10 Oct. 2003	12-15 Oct. 2004
Incubation begun	1 Nov. 2003	27 Oct. 2004
First harvest (n)	15 Feb. 2004 (12)	15 Dec. 2004 (5)
Second harvest (n)	18 May 2004 (12)	4 Feb. 2005 (5)

The IER analyses for NO_3^--N and NH_4^+-N were normalized to the mass of dry soil in each core and added to the post-harvest soil NO_3^--N and NH_4^+-N analyses to determine the total mineral N present after incubation. The baseline NO_3^--N and NH_4^+-N concentrations were subtracted from the post-incubation analyses to determine net mineralization. The experimental design did not include measurement of NH_4^+-N volatilization or NO_3^--N denitrification losses. Also, the soils were refrigerated for 2 to 3 weeks in October before the cores were ready for incubation. Thus, net mineralization results presented here are conservative and actual cool season net mineralization rates are probably greater. Student's t-tests were calculated in Microsoft Excel 2002.

RESULTS AND DISCUSSION

In all of the fields and in both years, the residual mineral N concentrations in October were greater than the 20 ppm specified as a goal in the Washington NRCS Nutrient Management Standard (USDA-NRCS, 2002). Table 4.3 presents net mineralization. Mean net mineralization from the beginning of incubation to 200 GDD (February) averaged 21.9 and 24.4 ppm in 2003-2004 and 2004-2005, respectively. Four of the seven soils mineralized greater than 20 ppm net N by 200 GDD. The low net mineralization value for Field E from 1 November 2003 to 15 February 2004 may have been due to denitrification. Water ponded in these cores and the soil appeared gleyed and mottled, indicating oxidation-reduction potentials suitable for denitrification.

Table 4.3. Mean net mineralization.

Field	1 Nov 2003 - 15 Feb 2004	1 Nov 2003 - 18 May 2004	27 Oct 2004 - 15 Dec 2004	27 Oct 2004 - 4 Feb 2005
	---------- ppm N (S.E.) ----------			
A	19.9 (2.7)*	27.2 (2.4)**	5.1 (7.0)	4.7 (6.6)
D	35.4 (9.7)	98.2 (12.5)**		
E	6.7 (9.6)	23.8 (9.0)*		
B			10.8 (10.8)	33.8 (5.8)*
F			4.4 (8.1)	37.2 (13.4)**
G			-2.9 (16.5)	22.1 (14.2)*
Mean	21.9 (4.8)	52.1 (8.0)**	4.3 (5.3)	24.4 (5.7)**

* post-incubation mineral N significantly greater than baseline mineral N at p = 0.10

** post-incubatoin mineral N significantly greater than baseline mineral N at p = 0.01

Distribution of mineral N for Field D cores in 2003-2004 is displayed in Fig. 4.1. In the fall, almost all mineral N was present as NO_3^-. By 200 GDD (15 February 2004), most of the mineral N was in the form of NO_3^- that had leached into the IER. By 18 May 2004, soil NO_3^- concentration had begun to increase again as temperatures warmed and precipitation decreased. These results are consistent with field observations of NO_3^- loss from Pacific Northwest cropped soils west of the Cascade Mountains in fall and winter (Zebarth and Paul, 1997).

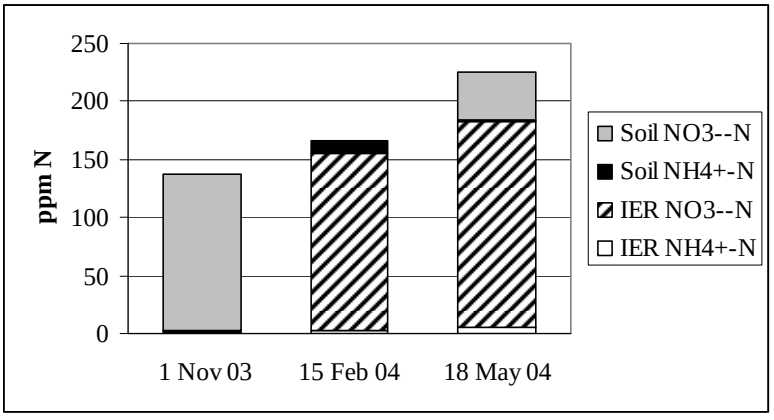

Fig. 4.1. Distribution of mineral N in Field D cores. Although the total mineral N was higher for this field than others in the study, the partitioning of mineral N was typical of all fields.

Fig. 4.2 compares net mineralization (beginning incubation to 200 GDD in February) to a mineralizable N index before incubation begins. The mineralizable N index was determined by the OSU Central Analytical Lab in Corvallis using anaerobic 7 day incubation at 40°C (Horneck et al., 1989). This is a modification of a method described by Keeney (1982). The mineralizable N index has been useful in predicting N mineralization to aid fertilizer decisions for winter wheat. However, the mineralizable N index had no value in predicting mineralization rates from the beginning of core incubations in late October or early November through 200 GDD in February in either year of this study.

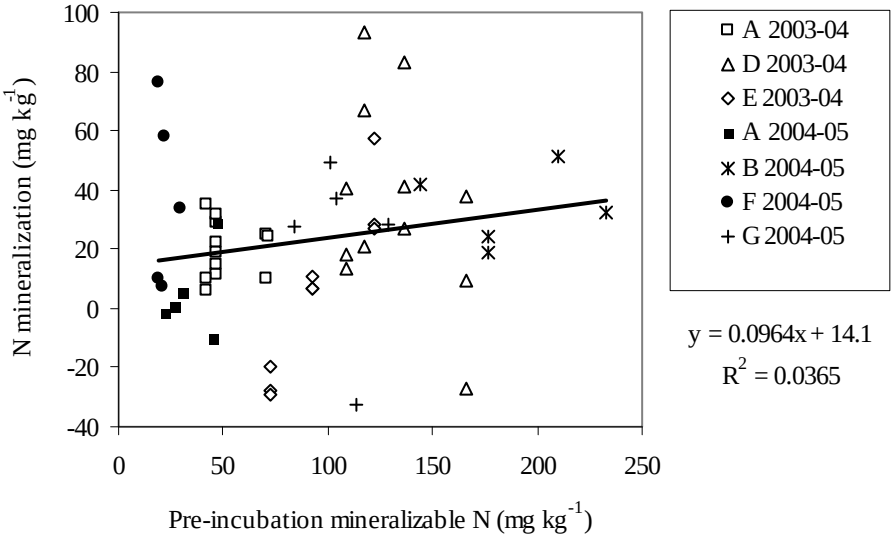

Fig. 4.2. N mineralization from beginning of incubations to 200 GDD in February verses the Oregon State University Central Analytical Lab mineralizable N index (Horneck et al., 1989) before incubations began.

CONCLUSIONS

All fields tested had residual NO_3^--N in excess of 20 ppm. Essentially all residual soil NO_3^- was lost from the cores by 200 GDD, indicating that this NO_3^- has the potential to negatively impact the environment.

This study defined "cool season" in Western Oregon as October to February when winter crops are present and October to May otherwise. Because there is little uptake of mineral N by plants during the cool season, N mineralized during this period has the potential to be lost to the environment. There was net mineralization of organic N by February on all soils tested in this study. In four of the seven soils tested, mineralization during this period added more than 20 mg N kg^{-1} soil. Additional mineralization occurred between February and May. The N mineralization measured in this study was a conservative estimate of total N mineralization under field conditions because volatilization and denitrification were not measured and the incubations were started in late October or early November. Thus, this study demonstrates that cool season N mineralization can contribute a significant mass of NO_3^- to the soil at a time when that NO_3^- is vulnerable to loss via leaching, runoff and denitrification.

In the fall, most of the mineral N in each core was present as NO_3^- in the soil. By late winter, most of this NO_3^- as well as most of the newly mineralized N had leached to the IER

below the soil. Between February and May, N mineralized in all soils tested and much of this mineralized N remained in the soil rather than leaching to the IER. This suggests that mineralization from February to May in these soils is less likely to contaminate groundwater than mineralization from October to February. The organic N mineralization rate in the cool season was not correlated to the fall mineralizable N index, suggesting that this index would not provide a useful measure of the expected extent of cool season recalcitrant organic N mineralization in manured soils.

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OF THE

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MANAGEMENT CONFERENCE

Volume 7

MARCH 8-9, 2007
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