Clinoptilolite Zeolite Influence on Nitrogen in a Manure-Amended Sandy Agricultural Soil

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Zeolite minerals may improve nitrogen availability to plants in soil and reduce losses to the environment. A study was conducted to determine the influence of clinoptilolite (CL) on nitrogen (N) mineralization from solid dairy manure (224 kg N ha^{-1}) in a sandy soil. Clinoptilolite was added to soil at six rates (0 to 44.8 Mg CL ha^{-1}), each sampled during 11 sampling dates over a year. Over time, nitrate (NO$_3$)-N increased, ammonium (NH$_4$)-N decreased, but total inorganic N increased. Clinoptilolite did not influence the nitrification rates of initial manure NH$_4$-N or mineralization of organic N (ON) over time. It is possible that adsorption of manure-derived potassium (K) outcompeted the NH$_4$-N for CL exchange sites. The ON concentration was constant up to 84 days and then decreased by approximately 18% over the remaining time of the study across all treatments. Clinoptilolite use in this sandy soil did not alter mineralization of N from dairy manure.

Keywords Ammonium, clinoptilolite, mineralization, nitrate, nitrification, nitrogen, zeolite

Introduction

Nitrogen (N) from manure can be used to optimize and sustain crop production in many agronomic systems, but nitrate-nitrogen (NO$_3$-N) derived from manure through mineralization is susceptible to leaching and denitrification loss from soils. Nitrogen losses due to leaching and denitrification from agricultural land, especially in sandy soils, are an environmental and economic concern (Perrin et al. 1998). Nitrogen loss from irrigated cropland can significantly contribute to nitrate levels in surface and groundwaters and can subsequently lead to waterway impairment and eutrophication. On a national scale, agriculture accounts for 50% and 60% of impaired lakes and rivers, respectively (U.S. EPA 2006). Thus, preventing offsite nutrient movement and increased crop N utilization is important.

Interest has increased in the use of zeolite minerals as a means of increasing N-use efficiency and lessening offsite N movement. Zeolites are naturally occurring aluminosilicate minerals (Kithome et al. 1998). More than 50 different types of zeolite minerals have been found (Tsitsishvili et al. 1992). Clinoptilolite (CL), one zeolite of interest, is composed of tectosilicates with isomorphous substitution of aluminum (Al^{+3}) and silicon (Si^{+4}). It has a high cation exchange capacity (CEC; Perrin et al. 1998) because the CL structure consists of layered and interlinked four- and five-tetrahedral rings, between which

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are open eight- and 10-tetrahedral ring channels (Vaughan 1978). This structure leads to a CL surface area, as high as 9.25×10^5 m^2 kg^{-1} (Kithome et al. 1998), and high CEC of 220 cmol kg^{-1} (Ming and Mumpton 1989). Furthermore, the CL structure is highly selective for potassium (K$^+$) and ammonium (NH$_4^+$) and less selective for sodium (Na$^+$) and divalent cations such as calcium (Ca^{+2}) (Perrin et al. 1998). Theoretically, the affinity of CL for NH$_4^+$ can potentially protect NH$_4^+$ from microbial access and thus reduce nitrification rates.

Zeolites have been shown to influence soil and plant N dynamics, manure nutrient characteristics, and animal nutrition. Research has shown increased growth and/or yield of a variety of crops because of either an effect of zeolites on improved N-use efficiency or reduced NH$_4^+$ toxicity (Huang and Petrovic 1994; Minato 1968; Torri 1978; Moore, Lewis, and Goldsberry 1984; Stoilov and Popov 1984). Research evaluating the effect of CL on NO$_3$-N leaching through soil have also been reported (Tarkalson and Ippolito 2010; Huang and Petrovic 1994; MacKown and Tucker 1985). Zeolites have been shown to reduce the loss of NH$_4$-N when mixed with bovine manure during composting and land application (Febles et al. 1991). Gamze Turan (2009) and Mahimairaja et al. (1994) found that zeolite added to composting poultry litter significantly reduced NH$_3$ losses compared to composting poultry litter without zeolite. However, research assessing effects of zeolites on N dynamics in soils following application of animal manures has been limited.

Thus, the purpose of the current study was to determine the influence of zeolite added to dairy manure on the quantity of inorganic (IN) and organic N (ON) in a common sandy agricultural soil in the Pacific Northwest (USA) over time in a controlled growth chamber environment. This study serves as a baseline to determine if potential CL research in large-scale field studies is warranted.

Materials and Methods

Soils and Clinoptilolite

A Wolverine sand (mixed, frigid Xeric Torripsamment) was collected from a depth of 0 to 30 cm in an agricultural field near Firth, Idaho. This soil is found in row crop production areas of southern Idaho and Oregon and occupies approximately 11,000 ha (NRCS 2009a). Soils similar to the Wolverine series (Xeric Torripsamment soils) occupy more than 5 million ha from the western United States to the Great Plains (NRCS 2009a). After collection, the soil was air dried and passed through a 5-mm sieve. Soil NO$_3$-N and NH$_4$-N concentrations were determined using the method of Mulvaney (1996). The Wolverine soil contained 15.5 mg NO$_3$-N kg^{-1} and 1.6 mg NH$_4$-N kg^{-1}. The cation exchange capacity of the Wolverine soil in the surface 0 to 30 cm is 4.5 cmol(+) kg^{-1} (NRCS 2009b). Clinoptilolite was obtained from the Zeocorp LLC mine located in Hines, Ore. Detailed properties of the CL are found in Table 1.

Solid Manure Collection and Analysis

Fresh dairy manure was collected from a local dairy in south central Idaho. The manure was transported to the laboratory, air dried, and ground (2 mm) for analysis. Total N (TN) was determined by combusting a 50-mg sample using a FlashEA1112 CN analyzer (CE, Elantech, Lakewood, N. J.). Manure NO$_3$-N and NH$_4$-N concentrations were determined using the method of Mulvaney (1996). Measured properties of the soil and manure are presented in Table 2.

Table 1
Selected properties of clinoptilolite used in the study

Property	Quantity
CEC [cmol(+) kg^{-1}]	155
Charge density [cmol(+) A^{-2}]	10.1×10^{-23}
Bulk density (g cm^{-3})	0.76
pH	7.5–8.0
Pore size (nm)	0.5
Pore volume (%)	51
Permeability (m s^{-1})	10^{-3}
Si (%)	31.9
Al (%)	6.09
Na (%)	3.03
K (%)	2.77
Fe (%)	1.12
Ca (%)	0.29
Mg (%)	0.26
NH_4-N (mg kg^{-1})	6.1
NO_3-N (mg kg^{-1})	2.2

Table 2
Soil and dairy manure source potassium and nitrogen contents (mg kg^{-1})

Source	Total K	Total N	NO_3-N	NH_4-N	IN[a]	ON[a]
Wolverine sand	981.5	291.6	15.5	1.6	17.1	274.5
Solid dairy manure	27,500	16,771	11.3	107.1	119.0	16,652

[a]IN = NO_3-N + NH_4-N; ON = Total N – IN.

Growth Chamber Pot Studies

Treatments consisted of dairy manure applied at a constant rate and CL at rates of 0, 6.7, 13.4, 20.2, 26.9, and 44.8 Mg CL ha^{-1} added to the Wolverine soil, both on a soil mass basis (0, 1.2, 2.4, 3.6, 4.8, and 8.0 g CL pot^{-1}). Manure was applied at a total N rate of 747 kg ha^{-1} and an estimated first growing season plant-available N rate of 224 kg N ha^{-1} (30% of total N [Bary, Cogger, and Sullivan 2004]). The manure N application rate was calculated based the University of Idaho N recommendations for Russet Burbank potatoes (Stark, Westermann, and Hopkins 2004). The quantity of NO_3-N plus NH_4-N added to each soil with CL applications was small compared to the N added in manure; the NO_3-N plus NH_4-N applied in the CL to the soil represented from 0.03 to 0.16% of the plant-available N applied in the manure sources. The total mass of soil, manure, and zeolite was 400 g for all treatments. Each treatment was replicated four times. Eleven sample dates (7, 14, 21, 28, 42, 56, 84, 177, 267, and 365 days after study initiation) of all treatments and replications were established (6 CL rate treatments × 4 replications × 10 harvest dates = 240 pots). All treated soils were homogenized by mixing in plastic bags (0.05 mm thick). The treated soils (still in the bags) were then placed in plastic pots (400 cm^3). The top of the plastic bags were folded over the outside of the pots to expose the surface of the treated soils

to the atmosphere. Each treatment and replication combination was organized in a complete randomized design for each sample time and then placed in a growth chamber with a constant temperature of approximately 21°C. Once per week, reverse osmosis water was added to all pots to bring all the soils back to a uniform weight (approximately 70% of soil water content at field capacity). At sample times, soil from each pot was homogenized by mixing within plastic bags and a subsample was removed and weighed wet (approximately 13 g), extracted with 2 M potassium chloride (KCl), and analyzed for NO_3-N and NH_4-N (Mulvaney 1996) using a flow injection analyzer. A second subsample was taken from the treatment soil mixture, weighed wet, air dried, and reweighed to determine the gravimetric water content of the soil to report NO_3-N and NH_4-N concentrations on a dry-weight basis. The remaining soil mixtures were air dried, a 20-g subsample was pulverized using a Pica Blender Mill Model 2601 (Cianflone Scientific Instruments Corporation), and total N was determined by combusting a 50-mg sample using a FlashEA1112 CNH analyzer (CE, Elantech, Lakewood, N. J.).

Statistical Analysis

Analysis of variance was used to determine the effects of CL rate and sample time on N components in soil using a completely randomized factorial model from Statistix 8 (Analytical and Software 2003). Analysis of variance was used to analyze the effect of CL application rate on N components within each harvest date using a completely randomized model from Statistix 8. The Tukey's HSD test was used for mean separations. Significance for all analysis was determined at the $\alpha = 0.05$ probability level.

Results and Discussion

Main effects of CL application rate and sample time and their interaction on the concentration of total IN, NO_3-N, and NH_4-N in soil were statistically significant (Table 3).

Table 3
Analysis of variance of treatment main effects and interactions for soil IN, NO_3-N, NH_4-N, and ON concentrations

Variable[a]	Source	df[b]	P > F
IN	CL rate (CLR)	5	< 0.001
	Sample time (ST)	9	< 0.001
	CLR × ST	45	< 0.001
NO_3-N	CLR	5	< 0.001
	ST	9	< 0.001
	CLR × ST	45	< 0.001
NH_4-N	CLR	5	< 0.001
	ST	9	< 0.001
	CLR × ST	45	< 0.001
ON	CLR	5	0.0390
	ST	9	< 0.001
	CLR × ST	45	0.9699

[a]IN = NO_3-N + NH_4-N; ON = Total N – IN.
[b]df = degrees of freedom.

Table 4

Concentrations of IN, NO$_3$-N, NH$_4$-N, and ON in soils over time treated with different rates of CL

Variable[a†]	CL Rate	Days after study start										Mean
		7	14	21	28	42	56	84	177	267	364	
	Mg ha⁻¹						mg kg⁻¹					
IN	0	29.5	30.5	48.9 a[b]	43.8	69.5 a	66.0	71.7	91.8	122.3 ab	93.6 ab	66.8
	6.7	33.6	36.7	32.7 ab	55.3	73.4 a	64.6	56.9	105.5	103.8 abc	89.9 ab	65.2
	13.4	28.1	33.4	20.4 b	63.3	27.1 b	18.9	48.1	58.6	78.1 c	90.3 ab	46.6
	20.2	34.2	41.0	40.8 ab	52.1	73.1 a	58.2	49.7	89.0	85.8 bc	124.0 ab	62.5
	26.9	36.4	41.2	25.2 ab	52.4	18.4 b	47.0	39.4	73.3	89.4 bc	74.2 b	49.7
	44.8	35.9	39.8	38.8 ab	57.9	43.7 ab	65.3	83.2	128.4	132.8 a	128.5 a	75.4
	Mean	32.9	37.1	34.5	54.1	50.9	53.3	58.2	91.1	105.3	100.1	61.7
ANOVA							P > F					
CL rate		0.0799	0.2463	0.0147	0.5271	<0.001	0.2769	0.1647	0.1312	0.0028	0.0250	
NO$_3$-N	0	14.9 a	26.0	48.1 a	43.0	67.6 ab	65.8	71.2	91.7	121.7 ab	93.2 ab	64.3
	6.7	16.8 a	32.6	31.7 ab	54.7	72.1 ab	63.8	56.4	105.4	103.6 abc	89.0 ab	62.6
	13.4	6.3 c	30.3	17.6 b	62.0	25.6 c	17.9	47.4	58.5	76.9 c	89.7 ab	43.2
	20.2	14.4 a	35.5	39.5 ab	49.3	72.2 a	57.8	49.1	88.9	85.8 bc	122.9 ab	61.5
	26.9	12.8 ab	33.4	22.7 b	50.8	16.8 c	46.7	38.9	73.2	89.3 bc	73.1 b	45.8
	44.8	7.3 bc	32.1	37.1 ab	54.4	41.7 bc	63.8	82.2	128.2	132.6 a	127.3 a	70.7
	Mean	12.1	31.7	32.8	52.4	49.3	52.6	57.5	91.0	101.7	99.2	58.0
ANOVA							P > F					
CL rate		<0.001	0.4559	0.0093	0.5682	<0.001	0.2867	0.1672	0.1316	0.0029	0.0285	
NH$_4$-N	0	11.4 c	3.16	0.61	0.61	1.35	0.16	0.43	0.06	0.38	0.24	1.84
	6.7	13.3 bc	3.15	0.79	0.49	0.90	0.47	0.38	0.07	0.13	0.57	2.03

13.4	17.2 ab	2.38	2.09	1.01	1.09	0.70	0.49	0.10	0.78	0.44	2.63
20.2	15.5 bc	4.29	0.98	2.01	0.62	0.29	0.43	0.07	0.01	0.71	2.49
26.9	18.7 ab	6.04	1.90	1.20	1.13	0.23	0.35	0.06	0.10	0.73	3.05
44.8	22.5 a	6.06	1.30	2.61	1.46	1.01	0.73	0.10	0.15	0.84	3.67
Mean	16.4	4.2	1.3	1.3	1.1	0.5	0.5	0.1	0.3	0.6	2.62
ANOVA						$P > F$					
CL rate	0.5973	0.0541	0.0915	0.1802	0.9364	0.6448	0.4836	0.9908	0.1837	0.9723	
ON	405.0	503.4	480.1	446.4	434.9	448.7	416.4	372.7	316.2	405.6	422.9
0											
6.7	402.0	457.0	442.5	446.6	437.9	435.1	412.5	360.9	351.9	428.1	417.5
13.4	477.2	481.7	487.5	478.5	526.5	479.8	455.8	422.1	381.7	390.3	458.1
20.2	491.4	472.1	457.8	477.9	474.4	428.1	461.9	431.9	402.3	358.3	445.6
26.9	484.6	468.6	469.0	455.0	444.9	503.7	402.6	347.1	363.2	365.8	430.5
44.8	428.5	448.4	486.7	464.7	478.4	436.0	395.3	326.2	352.8	373.3	419.0
Mean	448.1	471.9	470.6	461.5	466.2	455.2	424.1	376.8	361.4	386.9	432.3
ANOVA						$P > F$					
CL rate	0.4168	0.9295	0.8926	0.5744	0.4660	0.7196	0.1371	0.1895	0.5145	0.7823	

[a]IN = NO_3-N + NH_4-N; ON = Total N – IN. [b]For each variable and day after study start, values with the same letter are not significantly different at the 0.05 probability level within each column.

In general, over time, IN and NO_3-N increased, NH_4-N decreased, and ON remained unchanged (Table 4). Changes in IN were associated largely with changes in NO_3-N over time; NH_4-N amounts were small enough not to influence IN changes. Although the CL application rate main effect and interaction were significant, there was no pattern in the data to suggest that CL influenced IN, NO_3-N, and NH_4-N soil concentrations over time. In a separate study, Tarkalson and Ippolito (2010) applied commercial N fertilizer and CL in the Wolverine sand, finding that CL conserved IN, likely due to NH_4^+ adsorption on to CL exchange sites, thus protecting NH_4^+ from microorganism assimilation. Other researchers (Ferguson and Pepper 1987; MacKown and Tucker 1985; Weber, Barbarick, and Westfall 1983) have suggested that CL offers NH_4^+ protection against microbial nitrification by adsorption in the CL mineral lattice.

However, in the current study, when manure was applied there was no influence of CL rate on concentration of NO_3-N, NH_4-N, and ON. The lack of response over time could have been due to the manure application adding sufficient K^+ to the soil to out-compete NH_4^+ for CL exchange sites. Dairy manures, on an average dry-weight basis, contain between 20,000 and 25,000 kg total K kg^{-1}(Barbarick 2004). Furthermore, K in manure occurs mostly in a soluble form (Crop Management Extension Group 2010), while manure NH_4^+ availability is time dependent, being released from the organic fraction via mineralization. Goto and Ninaki (1980) and Rahmani et al. (2004) showed that zeolite ion selectivity followed the order of $K^+ > NH_4^+ > Na^+ > Ca^{2+} > Mg^{2+}$, supporting the contention that manure K^+ outcompeted NH_4^+ for CL exchange sites. The manure in this study had a concentration of 27,500 mg total K/kg (Table 2), constituting an application rate of 220 mg K pot^{-1} (1,210 kg K ha^{-1}).

Ammonium N contributed 50% to the IN at day 7, dropped to 11% by day 14, and dropped to only 0.6% at day 365. This further supports our observation that NH_4^+ was not protected from microbial nitrification by adsorption in the CL mineral lattice. Organic N concentrations were constant from initiation to day 84. Organic N concentrations were reduced by approximately 18% from an average of 457 mg kg^{-1} (7 days to 84 days) to 375 mg kg^{-1} (177 days to 365 days). This corresponds to an estimated 1-year ON mineralization factor of approximately 18% in this study. However, this study did not include the influence of plant N uptake.

Conclusion

Clinoptilolite rate did not influence the nitrification rates of initial NH_4-N in manure added to soil or mineralization of ON in soil over a 1-year period. This is in contrast to other reported results, which showed that CL inhibits NH_4-N changes via adsorption in the CL mineral lattice, essentially making CL a form of slow-release N fertilizer. The lack of CL response to retain NH_4^+ was likely due to competitive adsorption from K^+, which was readily available, and in high concentrations in manure. In the Wolverine soil series, we do not recommend the use of CL as a method of altering N dynamics is soil receiving dairy manure.

Acknowledgment

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