### ORIGINAL PAPER

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# A rapid procedure for estimating nitrogen mineralization in manured soil

Received: 23 February 2000

**Abstract** A routine soil testing procedure for soil N mineralization is needed that is rapid and precise. Not accounting for N mineralization can result in the overapplication of N, especially in soils with a history of manure application. Our objectives were to compare results from a recently proposed rapid laboratory procedure with: (1) long-term N mineralization under standard laboratory conditions, and (2) actual forage N uptake from soil receiving dairy cattle (Bos taurus) manure in a 2-year field study. The rapid procedure is based on the quantity of CO<sub>2</sub>-C evolved during 24 h under optimum laboratory conditions following the rewetting of dried soil. Dairy cattle manure was surface applied beginning in 1992 at annual rates of 0, 112, 224, or 448 kg N ha<sup>-1</sup> to field plots on a Windthorst fine sandy loam soil (fine, mixed, thermic Udic Paleustalf) near Stephenville, Texas (32°N, 98°W). Results of the oneday CO2 procedure were highly correlated with soil N mineralized from samples collected in March of 1995 (P=0.004) and 1996 (P<0.001) and with forage N uptake (P < 0.001) both years of the study. Residual inorganic N in the same soil samples was poorly correlated with soil N mineralization and forage N uptake.

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**Keywords** Carbon mineralization · Nitrogen mineralization · Soil testing · Manure · Nitrogen uptake

## Introduction

Accurate prediction of the quantity of N that is mineralized from soil organic matter during a growing season would result in more efficient use of N fertilizers and manure and decrease the potential for surface and groundwater contamination. Several soil N mineralization procedures have been advocated (Stanford and Smith 1972; Keeney 1982), but these procedures generally are not suited for routine soil testing because of the lengthy time periods they require. The Stanford and Smith (1972) method also underestimates N mineralization in soils normally exposed to repeated wetting and drying cycles (Campbell et al. 1988) because it does not account for the flush of N mineralization that occurs when dry soil is rewetted (Birch 1958; Cabrera 1993).

The concept of decay series has been used to estimate N mineralized from animal manure over several cropping seasons. Methods used to estimate decay series are often indirect, usually involving N uptake by successive crops following manure application. Klausner et al. (1994) reported a decay series of 0.21, 0.09, 0.03, 0.03, and 0.02 for five growing seasons following a dairy cattle manure application in New York state. The first number in the series represents the fraction of N that is mineralized the first year, the second represents the fraction of residual organic N mineralized during the second year, and so on. Decay series estimates are often site specific, may not be sensitive to changing soil conditions, and require an accurate history of previous manure applications.

Computer programs are also available for predicting nutrient release from animal manures. Twelve computer programs evaluated by Thompson et al. (1997), however, used potential N availabilities from manures ranging from 0 to 100% in the year of application, with an average of 37%. Chang and Janzen (1996) experimentally estimated that 56% of N added in beef feedlot manure was mineralized in their study, while Sanderson and Jones (1997) estimated that 25% of added dairy cattle manure N was removed by coastal bermudagrass [Cynodon dactylon (L.) Pers.] in their research.

Residual inorganic soil N, predominantly NO<sub>3</sub>, has been a useful tool for predicting crop N needs in low rainfall areas, but has generally been less successful in more humid regions because of NO<sub>3</sub> losses via leaching and denitrification (Schmitt and Randall 1994). The pre-sidedress soil NO<sub>3</sub> test was developed by Magdoff et al. (1984) based on the premise that not sampling until corn (Zea mays L.) reaches a specified growth stage allows N mineralization and N losses to occur as long as possible before a N fertilizer decision is made. This test is widely used in the northeastern USA (Jokela 1989) and is being adopted in some midwestern states (Blackmer et al. 1991). Research in Maryland (Meisinger et al. 1992) and Pennsylvania (Fox et al. 1989), however, showed poor correlations between grain yield and in-season NO<sub>3</sub> values.

Blackmer et al. (1989) stated that reducing N-fertilizer input by identifying non-responsive fields was an important use of this test in Iowa. Soil  $NO_3$  content measures N available at the time of sampling and does not necessarily indicate the ability of soils to mineralize additional N, which can be significant in soils receiving manures or other organic wastes.

Organic N is mineralized because of organic-C mineralization, and CO<sub>2</sub> evolution accordingly has been studied as a predictor of soil N mineralization. Castellanos and Pratt (1981) demonstrated that C released as CO<sub>2</sub> during a 1-week aerobic incubation of ten manures was a satisfactory index for estimating manure-N availability in a 10-month greenhouse trial. They hypothesized that a 2- or 3-day incubation might provide an equally satisfactory relationship. Gilmour et al. (1985) suggested that CO2 evolution might predict net N mineralization from plant residues added to soil, while Gilmour et al. (1996) showed that a 7-day incubation of biosolids could be used to predict decomposition at >60 days. Franzluebbers et al. (1996a) recently reported that net soil N mineralization and soil microbial biomass were related to soil C mineralized as CO<sub>2</sub> in as little as 1 day. The coefficient of determination  $(r^2)$  for CO<sub>2</sub> evolved during the first day after rewetting dried soil and net N mineralization after 21 days was 0.85 for eight diverse soils.

The prediction of organic C and N dynamics from CO<sub>2</sub> evolved after rewetting dried soil has a strong theoretical basis. Moderate drying of soil kills a portion of the soil microbial biomass (Jenkinson 1966) as well as rendering a portion of soil organic matter mineralizable because of physical disturbance (Van Gestel et al. 1991). The flush of microbial activity soon after rewetting probably reflects the contribution of both soil microbial biomass and other active organic matter pools that are easily mineralizable. Elliot (1986) hypothesized

that drying/rewetting is one mechanism by which each soil N pool is replenished from successively more recalcitrant or physically protected N pools. Inubushi and Wada (1987) found that air-drying and rewetting soil not only increased the easily mineralizable soil N pool, but also increased the size of a more stable pool that mineralized more slowly. Most biologically-based procedures normally advocate the use of field-moist soil, an additional limitation for routine soil testing that commonly requires dried soil. A procedure that rapidly and precisely estimates N mineralization from dried soil would increase adoption by soil-testing laboratories.

The objective of our study was to determine relationships between the flush of  $CO_2$  evolved during 1 day following rewetting of dried soil, and potential soil N mineralization in the laboratory and forage N uptake in the field from manure-amended soil.

# **Materials and methods**

Field plot design and soil sampling

A dryland field experiment utilizing coastal bermudagrass and bermudagrass overseeded with wheat (Triticum aestivum L.) was established near Stephenville, Texas (32°N, 98°W) in May 1992 (Sanderson and Jones 1997). The soil was a Windthorst fine sandy loam (fine, mixed, thermic Udic Paleustalf) with pH approximately 6.5 and containing 120 g kg<sup>-1</sup> and 660 g kg<sup>-1</sup> of clay and sand, respectively, in the surface 7.5 cm. Mean soil organic C and total N in this same soil depth were 14.1 g kg<sup>-1</sup> and 1.3 g kg<sup>-1</sup> soil. The average annual air temperature is 18°C and annual precipitation averages 750 mm. The experimental design was a 2 (cropping system) × 4 (manure N rate) factorial within a randomized complete block with four replicates; berms separated the blocks to prevent the overland flow of applied amendments. Each plot was  $3 \times 6$  m. Dairy cattle manure (0, 112, 224, or 448 kg N ha<sup>-1</sup> year<sup>-1</sup>) was surface applied in four equal applications each year to the two cropping systems, beginning in February 1992 through 1996. Concentrations of N, P, and K in the manure averaged 20.0, 5.6, and 16.6 g kg<sup>-1</sup>, respectively. Five harvests were made in 1995, one from wheat and four from coastal bermudagrass. Forage was harvested 5 times from both bermudagrass and bermudagrass overseeded with wheat in 1996. A sickle-bar mower was used to harvest a 1×6-m strip at a 5-cm height from the center of each plot at each harvest.

Forage samples for chemical analysis were hand-clipped from each plot at each harvest to avoid contamination with manure, rinsed with deionized water, and dried at 55 °C for 48 h before grinding to pass a 1-mm mesh. Bermudagrass N concentration was determined using a near-infrared reflectance spectrometer (Shenk and Westerhaus 1991). N concentrations of wheat samples were determined by wet chemical procedures (Baethgen and Alley 1989). Forage N uptake was calculated by multiplying dry matter yield and N concentration.

Thirty soil cores (2.5 cm diameter, 0 to 7.5-cm depth) were composited monthly from each plot from February through July in 1995; this was repeated in 1996. Samples were dried in a forceddraft oven at 40 °C for 24 h and passed through a 5-mm sieve (Franzluebbers et al. 1996a). Only results for March samplings are reported because this month generally resulted in the highest correlations, and this is also the period that producers in this region would sample soils to allow sufficient time to apply additional fertilizers or manure prior to the spring growth of bermudarrass

#### Soil C and N mineralization

Soil C and N mineralization were determined from three 40-g subsamples moistened to 55% water-filled pore space and incubated in 1-l glass jars. C mineralization was determined from  $\rm CO_2$  trapped in 10 ml of 1 M KOH during 24 days of incubation at 25 °C (Franzluebbers et al. 1996a). Alkali traps were titrated with standardized HCl to a phenolphthalein endpoint (Anderson 1982). A vial containing 10 ml water was also placed in each jar to maintain humidity.

N mineralization was determined from soil inorganic-N concentrations at 0 and 24 days of incubation. Subsamples were oven dried at 60 °C for 24 h, and ground to pass a 2-mm sieve. A 7-g portion was shaken with 28 ml of 2 M KCl for 30 min, with the filtered extract analyzed for NH<sub>4</sub><sup>+</sup>-N and NO<sub>2</sub><sup>-</sup>-N plus NO<sub>3</sub><sup>-</sup>-N using autoanalyzer techniques and the modified indophenol blue (Technicon Industrial Systems 1977a) and Cd reduction methods (Technicon Industrial Systems 1977b), respectively. Residual inorganic soil N was defined as the sum of the extracted forms of N given above determined immediately prior to incubation.

# Statistical analyses

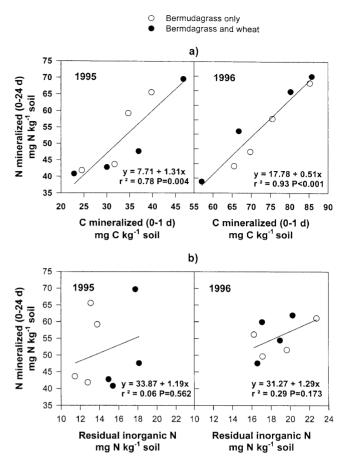
Linear regression and correlation were used to determine relationships among soil properties and forage N uptake (SigmaStat for Windows version 2.0, 1992–1995; Jandel, San Rafael, Calif.).

#### **Results and discussion**

C mineralized in 1 day from dried and rewetted soil was highly correlated with potential N mineralized in 24 days from soil samples collected in March 1995 and 1996 (Fig. 1a). March is the normal time for soil sampling for N recommendations for warm-season forages in this region, and residual soil NO<sub>3</sub> is the soil N test most commonly utilized. Wheat overseeded into bermudagrass and actively growing during soil sampling did not significantly affect the observed relationships (Fig. 1a).

Residual inorganic soil N was poorly correlated with potential N mineralized in 24 days for either year (Fig. 1b). Since mineralization of added manure N should be a principal source of residual inorganic N in this study, it was thought that residual inorganic soil N should correlate with N mineralized in laboratory incubations. Residual inorganic N from early spring soil samples, however, apparently would not be an adequate estimator of potential N mineralization in this manured soil.

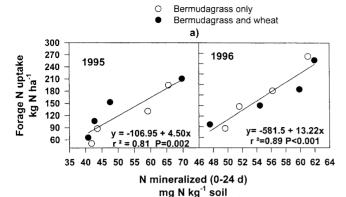
Soil N mineralized in 24 days in the laboratory in both study years was highly related with forage N uptake in the field (Fig. 2a). This procedure is too time consuming, however, to be used as a routine soil test. Forage N uptake both years was also very highly correlated with C mineralized in 1 day from dried and rewetted soil (Fig. 2c). The 1-day C mineralization procedure is sufficiently rapid to be used as a routine soil test and explained a slightly greater proportion of the variation in crop N uptake than did potential N mineralization. Slopes of the regressions for C mineralized in 1 day after rewetting dried soil vs. forage N uptake were very

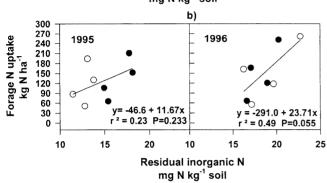


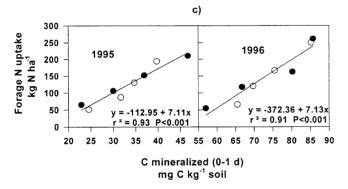
**Fig. 1** Relationships of soil N mineralized in 1995 and 1996 with **a** C mineralized in 1 day from dried and rewetted soil, and **b** soil residual inorganic N

similar for both study years, whereas slopes for 24-day soil N mineralization vs. forage N uptake varied three-fold between years. Wheat overseeded into bermudagrass vs. bermudagrass only did not influence the observed relationships. Residual inorganic soil N was poorly correlated with forage N uptake both years (Fig. 2b).

Thicke et al. (1993) reported that 1 week of aerobic incubation for N contributed as much as a 12-week incubation to models of corn grain yield and total N uptake as determined by stepwise multiple regression. Acid permanganate-, autoclave-, and glucose-extractable N and anaerobic incubation did not consistently contribute to the models. The authors found, however, that although the initial experiments with aerobic incubation resulted in promising relationships, results of field validation experiments were not reliably predicted probably because of yearly weather variation. Other authors have reported moderate correlations between results of chemical tests that extract a fraction of soil organic N and plant N uptake in the greenhouse or soil N mineralization in the laboratory (Keeney and Bremner 1966; Lathwell et al. 1972), but poorer results under field conditions (Fox and Piekielek 1978).







**Fig. 2** Relationships of forage N uptake in 1995 and 1996 with **a** soil N mineralized in 24 days, **b** soil residual inorganic N, and **c** C mineralized in 1 day from dried and rewetted soil

The flush of CO<sub>2</sub> in 1 day after rewetting dried soil appeared to adequately represent the contribution of the active soil microbial biomass and soil organic matter pools that were readily mineralizable (Franzluebbers et al. 1996b), based on relationships with laboratory N mineralization and forage N uptake. Partial desiccation of microbial biomass due to drying and rewetting and subsequent release of the desiccated microbial biomass as CO<sub>2</sub> may have contributed to these results. The Windthorst soil is naturally exposed to temperatures of >40 °C, the drying temperature used in this study, during summer months. Unpublished data from our laboratory shows only slightly greater C mineralization (2-3%) from soils dried at 40 °C vs. continuously moist soils, and the values are highly correlated  $(r^2>0.90)$ . Therefore, the flush of CO<sub>2</sub> following the rewetting of

soil dried at 40 °C may mimic the natural CO<sub>2</sub> flush of soil in the field from the partial desiccation and release of microbial biomass.

In summary, quantities of CO<sub>2</sub>-C evolved during the first day after rewetting dried soil were closely related to longer-term soil N mineralization and forage N uptake from soil receiving dairy cattle manure. Because of its relative simplicity, rapidity, and reliability, we recommend that this procedure be considered as a rapid test to estimate potential net N mineralization in manure-amended soils.

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