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Comparison of ^{15}N labelling methods to measure gross nitrogen mineralisation

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Abstract

A laboratory study was conducted to compare methods of labelling the soil NH_4^+ pool with ^{15}N as a prerequisite to the measurement of gross N mineralisation. Composite soil (0–12 cm) was collected from 12 sites differing in land use and soil texture. Three methods were used to apply ^{15}N to the soil: (i) addition of a $^{15}NH_3$–air gas mixture to the headspace above the soil (gas jar), (ii) injection of a $^{15}NH_3$–air gas mixture into re-packed soil cores (gas injector) and (iii) addition of a $(^{15}NH_4)_2SO_4$ solution pipetted onto the soil surface (solution). ^{15}N isotopic dilution was determined between 24 and 72 h after ^{15}N application. Of the applied ^{15}N 52 to 93% was recovered as inorganic N 24 h after application. Less ^{15}N was recovered in the clay loam compared to the sandy loam and silty clay loam for all methods. Recovery from the gas injector was lower than from the gas jar or solution treatments in the sandy loam and silty clay loam. No difference in the recovery of applied ^{15}N between methods was observed in the clay loam. ^{15}N isotopic dilution was less in the gas jar compared to the gas injector and solution treatments. This resulted in significantly lower estimates of gross N mineralisation rates and NH_4^+ consumption for the gas jar treatment. The non-uniform distribution of ^{15}N through the soil probably caused these differences as a consequence of $^{15}NH_3$ dissolving quickly in the surface of the soil. In seven of the 12 soils, gross rates of N mineralisation were not significantly different when ^{15}N was applied as solution or by gas injection. In the remaining five soils, estimates of gross N mineralisation rates were greater in the solution treatment. Ammonium consumption was significantly different between the solution and gas injector treatments in three soils. Where there were differences in values for gross N mineralisation and NH_4^+ consumption rates between methods, the effects were not associated with a particular soil texture or land use. Greater nitrification of $^{15}NH_4^+$ to $^{15}NO_3^-$ and shorter pseudo-residence times of the NH_4^+ pool were observed in the solution treatment compared to gas injector. This suggests a difference in the distribution of ^{15}N within soil microsites, with the ^{15}N applied in the solution treatment being more accessible for microbial consumption. Nevertheless, application of ^{15}N as an NH_4^+ solution, or injection of $^{15}NH_3$ gas into soil, resulted in estimates of a similar magnitude for gross N mineralisation rates and identified the same differences between soil type and land use. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: ^{15}N labelling methods; ^{15}N isotopic dilution; NH_3 gas injector; Gross nitrogen mineralisation; Ammonium consumption; Ammonification; Soil nitrogen supply; Pseudo-residence time; Agricultural soils

1. Introduction

A prerequisite for determining gross nitrogen mineralisation rates by ^{15}N isotopic dilution methods is the uniform labelling of the soil ammonium pool with

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Table 1
Soil characteristics in the 0–12 cm soil layer

Site	Land use	Total N (%)	Total C (%)	Soil C-to-N ratio	pH
Woburn	arable	0.10	0.73	7.30	7.0
	ley 2 yr	0.21	1.69	8.05	6.9
	ley 6 yr	0.21	2.02	9.62	6.9
	pasture	0.24	3.42	14.25	5.4
Rosemaund	arable	0.19	1.47	7.74	6.6
	ley 2 yr	0.25	2.07	8.28	6.4
	pasture	0.41	3.95	9.63	5.6
	reseed	0.48	3.84	8.00	6.0
North Wyke	grass–clover-d	0.56	6.58	11.75	5.5
	grass-d	0.63	8.18	12.98	5.5
	reseeded grass	0.72	8.46	11.75	5.5
	grass-u	0.86	9.61	11.17	5.3

^{15}N. In laboratory experiments, this has typically involved the application of ^{15}N-labelled salt solutions to disturbed soil at near optimal soil water contents (Nishio et al., 1985; Myrold and Tiedje, 1986; Bjarnason, 1988). Mixing ^{15}N-labelled solution into the soil ensures uniform distribution, but disturbance may affect mineralisation rates through enhanced microbial activity (Cabrera and Kissel, 1988; Sierra, 1992). It is difficult to apply ^{15}N uniformly without disturbance. Application of ^{15}N solution to the soil surface gives a uniform distribution if the soil depth is restricted to a few centimetres (Crawford and Chalk, 1992; Pilbeam et al., 1993; Barraclough and Puri, 1995; Willison et al., 1998). This approach reduces any possible 'flush' in microbial activity associated with mixing of the soil immediately prior to incubation. Alternatively, the use of injectors to distribute ^{15}N-labelled solution into soil cores enables in situ rates of gross N mineralisation to be determined with minimal soil disturbance (Davidson et al., 1991; Ambus et al., 1992; Monaghan, 1995; Sparling et al., 1995; Ledgard et al., 1998). However, multiple injections within each soil core are required to ensure a uniform distribution (Monaghan, 1995; Sparling et al., 1995).

As well as the direct effects of disturbance on aeration, the addition of ^{15}N-labelled solution increases the water content which may stimulate microbial activity and solutions should not be used when the soil is dry (Davidson et al., 1991; Stark and Firestone, 1995). Sparling et al. (1995) also suggested that the use of solutions be restricted to heavier textured soils. Methods used to enrich the soil with ^{15}N without the addition of water have been developed to measure gross N mineralisation (Murphy et al., 1997) and gross nitrification (Stark and Firestone, 1995; Willison et al., 1998). Increasing the ^{15}N-enrichment of the soil NH_4^+ pool by the injection of dilute mixtures of ^{15}N-labelled ammonia gas in air has proved successful for use within coarse textured soils with low organic matter content (Murphy et al., 1997, 1998a). However, this method has not been tested on heavier textured soils. Stark and Firestone (1995) have shown that ^{15}N label can be introduced into the soil nitrate pool (to measure gross nitrification) by adding ^{15}N-labelled nitric oxide (NO) into the headspace above soil contained within 1 L glass jars. Although not applicable for use with undisturbed soil cores the addition of ^{15}N labelled gas into a headspace above soil has the advantage of providing a simple experimental design that does not require purpose-built equipment or the use of solutions.

To date no consideration has been given to the possible effect that differences in methods of ^{15}N application may have on calculated gross rates of N mineralisation. Therefore a comparison of the methods used to create a uniformly enriched pool of NH_4^+, and an assessment of their efficacy, is both important and timely. Our aim was to determine if application of ^{15}N to soil by: (i) addition of a $^{15}NH_3$–air gas mixture to the headspace above soil (gas jar), (ii) injection of a $^{15}NH_3$–air gas mixture into soil cores (gas injector) or (iii) pipetting a $(^{15}NH_4)_2SO_4$ solution onto the surface of soil (solution) resulted in the same recovery of ^{15}N and rate of ^{15}N isotopic dilution when soil moisture content was near field capacity. This information was required to: (i) assess whether methodological differences are likely to affect the comparison of gross N transformation rates between experimental data sets, and (ii) to determine if the use of $^{15}NH_3$ was a suitable means of labelling the soil NH_4^+ pool in heavy textured soils. This paper is concerned with methodology; the biological significance of the gross N mineralisation rates in the contrasting soils and land uses will be discussed elsewhere.