

The fate of slurry-N fractions in herbage and soil during two growing seasons following application

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Abstract Farmers are under increasing pressure to use slurry-nutrients more efficiently in order to maximise crop utilisation and minimise losses to the environment. The objective of this field experiment was to quantify the fate of three N fractions (urine-N [U], rapid faecal-N [FR] and slow faecal-N [FS]) from cattle slurry in herbage and soil. The recovery of the three slurry-N fractions was measured in the first and second year after application on a permanent grassland in Ireland. Urine and faeces were collected from cows fed with ^{15}N -labelled herbage, or unlabelled herbage with added ^{15}N -labelled urea and these were recombined to produce differentially labelled experimental slurries. Slurries were applied to plots, and

^{15}N -enrichments of the herbage and three soil layers were determined. The initial recovery of ^{15}N (6 weeks after application) in herbage was 18%, 13%, 2%, while the residual recovery (12–63 weeks) was 4%, 6% and 7% for U, FR and FS, respectively. The total slurry-N recovery in the plant-soil system was estimated to range from 45% for urine-N to 72% for faecal-N. These results increase our mechanistic understanding of slurry-N dynamics in soil-plant systems and will inform models used to predict the fate of cattle slurry applied to grassland.

Keywords ^{15}N stable isotope tracer · Grassland · Cattle slurry · Residual nitrogen recovery · Faecal-N · Urine-N

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Introduction

Slurry is an important source of nutrients in grassland systems and farmers are under increased pressure to improve the utilisation of slurry nitrogen (N), especially in the European Union since the implementation of the EU Nitrates Directive (91/676/EEC) (Anon 1991) and Water Framework Directive (2000/60/EC) (Stark and Richards 2008). Knowing when slurry-N is taken up by herbage is important in order to maximise crop utilisation and minimise environmental degradation. Herbage uptake of slurry-N may be immediate, i.e. prior to the first herbage cut after application, or it

may be residual and taken up by the plant over a sustained period of time (Schröder 2005b; Thomsen 2005).

Approximately 50% of the N in cattle slurry is in an inorganic form (mainly ammonium-N [$\text{NH}_4\text{-N}$] originating from urea in urine) (Anon 2010) which is directly available for plant uptake after application. However, during and after application, large losses from this fraction (up to 80%) can occur due to ammonia volatilisation (Søgaard et al. 2002; Sommer and Hutchings 2001). These losses can be minimised by using low emissions application techniques (Misselbrook et al. 2002; Smith et al. 2000; Webb et al. 2010). Some 15–35% of $\text{NH}_4\text{-N}$ is also immobilised by the soil microbial biomass in the first weeks after application, and may be gradually mobilised over time (Jensen et al. 2000; Morvan et al. 1997). A large proportion of mineral N, derived from slurry, can also be lost through nitrate leaching and denitrification (Schröder 2005a).

The remaining 50% of the slurry-N is in an organic form (faecal-N) and needs to be mineralised prior to it becoming plant-available. Mineralisation of the organic slurry-N fraction is difficult to predict as it is affected by factors such as slurry composition, soil type, temperature, pH, aeration and soil moisture conditions (Calderon et al. 2005; Chadwick et al. 2000; Fanguero et al. 2008; Van Kessel and Reeves 2002). Labelling of animal manures with ^{15}N stable isotope tracers has been established as an important tool to assess N release because it has a number of important advantages over conventional agronomic methods (Cusick et al. 2006; Dittert et al. 1998). The results from ^{15}N labelling tend to be less variable, which is particularly important when attempting to quantify the relatively small residual N recoveries (Cusick et al. 2006; Muñoz et al. 2003; Thomsen 2004). Additionally, ^{15}N can be traced in both soil and plant (as well as leachate and gaseous emissions if required), providing valuable information about the fate of N not taken up by plants (Chadwick et al. 2001; Dittert et al. 2001; Morvan et al. 1997; Sørensen and Amato 2002; Thomsen et al. 1997). Also, with ^{15}N labelling the fate of urine and faecal-N from slurry can be monitored separately, offering a more detailed insight into the N dynamics of organic and inorganic-N fractions (Bosshard et al. 2009; Jensen et al. 1999; Sørensen et al. 1998; Thomsen et al. 1997).

Methods have been developed to label different faecal-N fractions (Powell et al. 2005; Powell et al. 2004), but there are some important pitfalls to consider. The main pitfalls for interpreting bulk ^{15}N values include the non-homogeneous ^{15}N labelling of faecal-N, which depends on the duration of feeding ^{15}N labelled forage to the animal, on the type of diet, and on the metabolism of the individual animal. Non-uniform labelling may result in an over- or underestimation of the mineralised proportion of faecal-N in the soil (Powell et al. 2004; Sørensen et al. 1994). Also, the increased microbial activity upon slurry addition may result in an exchange between non-labelled biomass N with inorganic ^{15}N , while the net amount of plant-available N may have remained constant (Dittert et al. 1998). This can lead to an over- or underestimation of the slurry-N recovery in the plant (Schröder 2005a).

Even though there have been many studies using ^{15}N labelling to trace the fate of manure N in arable systems (Dittert et al. 1998), we have not been able to identify any studies on grassland, with the exception of studies that focussed on the fate of ^{15}N labelled $\text{NH}_4\text{-N}$ (Chadwick et al. 2001; Dittert et al. 2001; Hoekstra et al. 2010; Morvan et al. 1997). Therefore, the objective of the present experiment was to trace the fate of three slurry-N fractions (urine-N, rapid faecal-N and slow faecal-N) in herbage and multiple soil layers in the first 62 weeks after a single slurry application on a permanent grass sward, using ^{15}N labelling techniques. This will provide us with better insights into the immediate and residual N uptake by grass from slurry, a better understanding of soil N cycling and a quantification of N losses from such grassland systems.

Materials and methods

Experimental design

The experiment was located at the Johnstown Castle Environment Research Centre, Wexford, Ireland, in a permanent perennial ryegrass-dominated grassland on a moderately drained, fine loamy soil that was not grazed and did not receive any N fertiliser or slurry during the year 2007 prior to treatment application. The experiment used 1 m × 1 m micro-plots confined with 10 cm high metal borders, which were placed into the soil to 5 cm depth (Fig. 1).

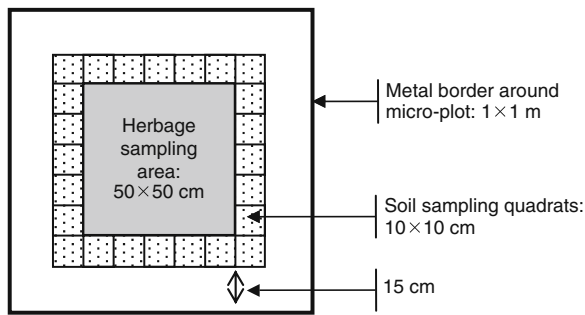


Fig. 1 Plot layout highlighting soil and herbage sampling areas

In order to facilitate both herbage and soil sampling without affecting herbage growth for subsequent harvests, the micro-plots were divided into a herbage sampling area and soil sampling area (Fig. 1). Herbage was sampled in the central 50 cm quadrat and soil samples were taken from a grid surrounding the 50 cm quadrat, but at least 15 cm from the micro-plot boundary. There was a 50 cm discard area between main plots. The plot layout was a complete randomised block design with five blocks.

The five treatments consisted of two control treatments (no slurry and non-labelled slurry) and three differentially labelled slurries, as described below and in Table 1:

- Urine-labelled slurry (ULS)
- Faeces-labelled slurry, Urea method (FLS_{UM})
- Faeces-labelled slurry, Herbage method (FLS_{HM})

Slurry ¹⁵N labelling and application

For the ¹⁵N labelling of faeces and urine, two non-lactating cows were used, which are preferred over milking cows in order to prevent 'loss' of expensive ¹⁵N in milk, even though the manure composition may be slightly different (Powell et al. 2004). Cattle slurry was labelled using two different, modified methods based on Powell et al. (2004): a) ¹⁵N labelled herbage was fed to cows in order to label the entire faecal-N pool, including undigested feed N, or b) feed urea ¹⁵N labelling was used to label the microbial fraction and the endogenous N of the faeces.

- a) For the ¹⁵N-herbage labelling method, a perennial ryegrass-dominated permanent sward was fertilised with double ¹⁵N labelled (10 atom%) ammonium nitrate (both the ammonium-N and nitrate-N were enriched) at a rate of 45 kg N ha⁻¹. The herbage was harvested after a 5-week regrowth period when the enrichment of the herbage at harvest time was approximately 3 atom%. Non-labelled herbage was produced in the same way, but using non-labelled fertiliser. This non-labelled herbage was used in the adaptation period, for the ¹⁵N-urea method (below), and to allow the continuation of similar feeding during the manure collection period after ¹⁵N herbage feeding had stopped.

For the ¹⁵N herbage-feeding method, the first cow was first adapted to the fresh grass diet for

Table 1 Components, chemical composition and isotopic enrichment of experimental slurries

Slurry type	Code	Slurry components		Slurry chemical composition			N content in slurry fractions (% of total slurry N) ^b			¹⁵ N enrichment (atom%)		P ¹⁵ N _{FS} ^c	
		Faeces ^a	Urine	DM (g kg ⁻¹)	N (g kg ⁻¹ DM)	NH ₄ -N (g kg ⁻¹ DM)	U	FR	FS	Slurry	FR		FS
Non-labelled slurry	NLS	NL	NL	68.5	81.2	45.3	59%	27%	14%	0.369	NA	NA	NA
Urine-labelled slurry	ULS	NL	L	69.2	91.2	56.0	62%	25%	13%	0.937	NA	NA	NA
Faeces-labelled slurry, Urea method	FLS _{UM}	L	NL	59.8	82.4	39.3	63%	25%	13%	0.563	0.987	0.522	10%
Faeces-labelled slurry, Herbage method	FLS _{HM}	L	NL	64.3	81.4	43.6	61%	26%	14%	1.140	2.261	1.590	25%

^a NL = non-labelled; L = labelled

^b U = Urine, FR = Faeces, rapid fraction (non NDF), FS = Faeces, slow fraction (NDF), NDF = Neutral Detergent Fibre

^c P¹⁵N_{FS} = proportion of the faecal excess ¹⁵N in the slow (NDF) fraction (Eq. 5a and 5b).

7 days, of which the last 2 days were used to collect unlabelled faeces and urine for recombination (see below). On day 8, the ^{15}N labelled herbage material was harvested and divided into 10 equal parts (to assure uniform ^{15}N feeding) on a weight basis. The grass was offered to the cow five times daily (approximately 15 kg DM [dry matter] day^{-1}) for 3 days.

- b) For the urea ^{15}N labelling method, 120 g urea per day (^{15}N at natural abundance) was sprinkled onto unlabelled grass fed to the second cow during the 7-day adaptation period. On day 8, single 24 g doses of 10 atom% ^{15}N urea mixed with water were sprinkled over the grass and fed to the cow five times daily for 2 days (10 doses per cow).

Total faeces and urine were collected at approximately 6-hour intervals after initial ^{15}N feeding up to at least 48 h after the ^{15}N feeding had stopped. Urine was collected by gluing a harness to the cows with a tube connection to drain the urine into a container. Faeces were collected from a crate on the floor. The collected faeces and urine were frozen until later use.

Labelled faeces were proportionally recombined with unlabelled urine, and vice versa, in order to make four different slurries (Table 1):

- NLS (non-labelled slurry),
- ULS (urine-labelled slurry),
- FLS_{UM} (faeces-labelled slurry, urea feeding method),
- FLS_{HM} (faeces-labelled slurry, herbage feeding method).

Tap water was added in order to obtain a DM content of approximately 7% in each slurry type.

Slurries were applied to the field micro-plots on 29th June 2007 at a rate of 3.3 kg m^{-2} by watering can, mimicking broadcast application by conventional slurry tankers.

Sampling methods

The plots were harvested five times during 2007 and 2008 (Table 2); the final harvest took place 62 weeks after slurry application. The herbage from the inner square of the micro-plots (Fig. 1) was harvested with electrical grass shears at 5 cm height, weighed and a sub-sample taken for DM, total N and ^{15}N analyses.

At each harvest, two soil cores (2 cm diameter) from each of three randomly selected grid-squares were taken to 25 cm depth (Fig. 1). The sampled grids were not revisited at later harvests. The samples were split into three depths: 0–5 cm, 5–15 cm and 15–25 cm and samples were immediately stored at 4°C.

Analytical procedures

Fresh soils were sieved through a 2 mm screen before further analyses. Slurry, grass and soil dry matter contents were determined by drying at 100°C overnight. Slurry and faecal total N were determined by Kjeldahl digestion of fresh slurries and faeces. $\text{NH}_4\text{-N}$ was extracted from fresh slurry by shaking 10 g of slurry in 200 ml 0.1 M HCl on a peripheral shaker for 1 h and filtering through a No. 2 Whatman filter paper. Concentrations of $\text{NH}_4\text{-N}$ in the filtrate were determined on an Aquakem 600 discrete analyser (Thermo Electron OY, Vantaa, Finland).

Table 2 Slurry application and plot harvest dates and weather data

Activity	Date	Weeks after appl.	Mean air temperature (°C)	Mean rainfall (mm day^{-1})	Cum. degree days (>5°C)	Mean SMD (mm) ^a	Mean effective drainage (mm day^{-1}) ^a
Slurry appl.	29/06/2007						
Harvest 1	13/08/2007	6	14.8 (1.0)	3.3 (4.9)	505	5.2 (5.5)	0.75 (2.1)
Harvest 2	24/09/2007	12	15.0 (2.0)	2.6 (5.4)	923	23.6 (17.3)	0.84 (2.6)
Harvest 3	19/03/2008	38	8.3 (2.9)	2.1 (3.7)	1532	3.3 (6.0)	1.13 (2.6)
Harvest 4	02/07/2008	53	10.8 (3.2)	2.8 (6.7)	2141	10.5 (7.2)	0.71 (3.9)
Harvest 5	08/09/2008	62	15.1 (1.4)	5.2 (8.5)	2825	7.8 (14.5)	3.00 (5.2)

^a Calculated using the soil moisture model by Schulte et al. (2005). SMD, Soil Moisture Deficit

Values in brackets indicate 1 standard deviation

Faecal neutral detergent fibre (NDF) was extracted from a freeze dried sub-sample according to the method by Van Soest et al. (1991) (Dungait et al. 2005) and the total N in the NDF fraction (NDFN) was measured using a NA1500 automated nitrogen-carbon analyser (Carlo Erba, Milan, Italy) coupled to a 20/20 isotope ratio mass spectrometer (SerCon Ltd, Crewe, UK).

Inorganic-N, $\text{NH}_4\text{-N}$ and total oxidised-N (TON i.e. NO_3 and NO_2) in fresh soil were determined by extraction in 2 M KCl (40 g soil: 100 ml KCl, shaken for 1 h), with $\text{NH}_4\text{-N}$ and TON determined on an Aquakem 600 discrete analyser.

Soil microbial biomass was extracted from the soils using the fumigation-extraction (FE) method (Sparling and West 1988). Ten grams of field moist soil were extracted with 40 ml of potassium sulphate (0.5 M K_2SO_4), either directly or after 24 h of chloroform fumigation. The samples were shaken for 1 h and filtered. Soil microbial biomass N (SMB-N) was determined in the extract by persulphate digestion (Cabrera and Beare 1993) using a conversion factor of $k_{\text{eN}}=0.54$ (Brookes et al. 1985).

A composite sub-sample of the dried grass was milled through a 2 mm screen and subsequently ground to a fine powder in a ball mill. Total N and ^{15}N concentrations in the dried and milled grass and soil samples were measured on an ANCA 20/20 SL combustion isotope ratio mass spectrometer (IRMS) (Delta plus, Finnigan, Bremen, Germany). The ^{15}N isotopic enrichments of total slurry-N, total faecal-N, soil inorganic-N and SMB-N were determined after diffusion of the Kjeldahl digest, KCl extract and persulphate digest, respectively (Stark and Hart

1996) and measured on an IRMS (Delta plus, Finnigan, Bremen, Germany). The ^{15}N isotopic enrichment of the faecal-NDFN was measured on a 20/20 isotope ratio mass spectrometer (SerCon Ltd, Crewe, UK).

Calculations and estimations of other pathways

^{15}N recovery of labelled fractions

For each of the three slurry labelling treatments (ULS, FLS_{UM} and FLS_{HM}), the percentage of slurry ^{15}N recovered in herbage (^{15}NRH) was calculated as:

$$^{15}\text{NRH}(\%) = \frac{H(c-d)}{S(a-b)} \times 100 \quad (1)$$

where H =total herbage N uptake, S =slurry-N applied, a =atom% ^{15}N of applied slurry, b =atom% ^{15}N of unlabelled slurry, c =atom% ^{15}N of herbage from labelled plots, d =atom% ^{15}N of herbage from control plots.

Similar equations were applied to calculate the ^{15}N recovery in the total soil ($^{15}\text{NRS}_T$), in the inorganic soil N fraction ($^{15}\text{NRS}_I$) and soil microbial biomass N ($^{15}\text{NRS}_{\text{SMB}}$).

While the recovery of ULS is a direct reflection of the slurry urine fraction, the two faecal labelling methods are more confounded. For FLS_{HM} , the total faecal-N is enriched, and therefore both the slow and rapid faecal fractions are labelled. In the FLS_{UM} labelled faeces, only the microbial and endogenous N fractions are enriched. In Fig. 2, we present two different theories, on the basis of which the contribution of both the rapid

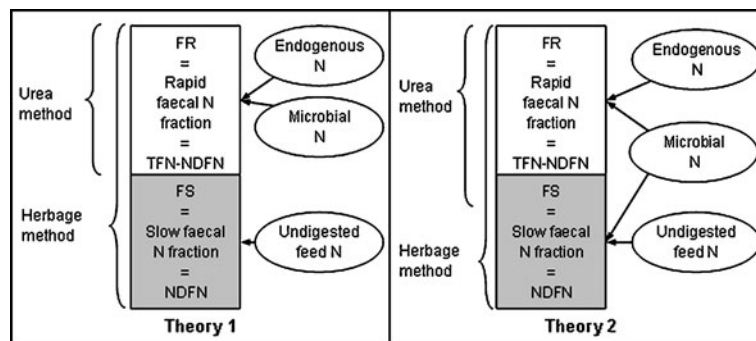


Fig. 2 Conceptual presentation of ^{15}N labelling of the slowly (FS) and rapidly (FR) degradable faecal-N fractions with the urea and herbage methods. Theory 1 is based on work by

Powell et al. (2004) and Theory 2 is based on Sørensen et al. (1994). Abbreviations: TFN=total faecal-N, NDF=neutral detergent fibre, NDFN=N in the NDF fraction

and slow faecal fractions to total ^{15}N recovery can be determined, as explained below.

Theory 1 is derived from the work by Powell et al. (2004), and is based on two main assumptions:

- The urea method labels the rapid faecal-N (FR) fraction only, which is equivalent to the non-NDFN fraction of the faeces
- The herbage method labels both the slow (FS, which is equivalent to NDFN) and rapid fraction of faeces, and the labelling is homogenous i.e. the enrichment of NDFN is similar to the enrichment of non-NDFN (or total faecal-N).

Therefore the recovery from the rapid faecal fraction ($^{15}\text{NR}_{\text{FR}}$) is equal to the recovery from the urea-labelled slurry:

$$^{15}\text{NR}_{\text{FR}} = ^{15}\text{NR}_{\text{FLS}_{\text{UM}}} \quad (2)$$

The ^{15}N recovery from FLS_{HM} is the sum of the recovery from the fast and slow faecal fractions multiplied by the proportion of the slow and rapid fractions in the total faecal-N, and can be represented by the following equation:

$$\begin{aligned} ^{15}\text{NR}_{\text{FLS}_{\text{HM}}} = & ^{15}\text{NR}_{\text{FLS}_{\text{UM}}} \times \left(1 - \frac{\text{NDFN}_{\text{HM}}}{\text{TFN}_{\text{HM}}}\right) \\ & + ^{15}\text{NR}_{\text{FS}} \times \frac{\text{NDFN}_{\text{HM}}}{\text{TFN}_{\text{HM}}} \end{aligned} \quad (3a)$$

which can be rewritten as:

$$\begin{aligned} ^{15}\text{NR}_{\text{FS}} = & \left(^{15}\text{NR}_{\text{FLS}_{\text{HM}}} - ^{15}\text{NR}_{\text{FLS}_{\text{UM}}} \times \left(1 - \frac{\text{NDFN}_{\text{HM}}}{\text{TFN}_{\text{HM}}}\right) \right) \\ & \times \frac{\text{TFN}_{\text{HM}}}{\text{NDFN}_{\text{HM}}} \end{aligned} \quad (3b)$$

in which $^{15}\text{NR}_{\text{FLS}_{\text{HM}}}$, $^{15}\text{NR}_{\text{FLS}_{\text{UM}}}$ and $^{15}\text{NR}_{\text{FS}}$ are the ^{15}N recovery of the herbage-labelled faecal-N (both slow and rapid fractions), urea-labelled faecal-N (rapid fraction only) and slow faecal-N fraction, respectively. NDFN is the N in the NDF fraction (equivalent to slow faecal-N) and TFN is total faecal-N.

However, both assumptions in Theory 1 can cause considerable problems. Firstly, it is notoriously difficult to achieve homogenous labelling of the

whole faecal fraction (Powell et al. 2004; Sørensen et al. 1994). Secondly, work by Sørensen et al. (1994) suggests that the divide between the slow and rapid fractions is not between undigested feed N and the remaining faecal-N, but that some of the microbial faecal fraction (microbial N from the rumen) can also be considered to be slowly available. This would suggest that the urea feeding method not only labels the rapidly available pool, but also part of the slowly available pool (Fig. 2, Theory 2). Therefore, we propose Eq. 4 to describe the enrichment of the UM and HM labelled faeces as a function of the enrichments of the slow and rapid faecal fractions (NDFN and non-NDFN) and their proportion in total faecal-N:

$$\delta^{15}\text{N}_{\text{TF}} \times \text{TFN} = \text{FS} \times \delta^{15}\text{N}_{\text{FS}} + \text{FR} \times \delta^{15}\text{N}_{\text{FR}} \quad (4)$$

in which $\delta^{15}\text{N}_{\text{TF}}$, $\delta^{15}\text{N}_{\text{FS}}$ and $\delta^{15}\text{N}_{\text{FR}}$ are the ^{15}N atom% excess of the total faecal-N, the slow faecal-N (NDFN) and the rapid faecal-N (non-NDFN) (excess ^{15}N refers to the absolute ^{15}N enrichment minus the natural abundance, which was 0.369%) and TFN, FS and FR refer to the N content of the total faeces, and the slow and rapid faecal-N fractions, respectively. Therefore the proportion (P) of the total ^{15}N excess in faeces that can be attributed to the slow fraction ($P^{15}\text{N}_{\text{FS}}$) and fast fraction ($P^{15}\text{N}_{\text{FR}}$) is calculated as:

$$P^{15}\text{N}_{\text{FS}} = \frac{(\delta^{15}\text{N}_{\text{FS}} \times \text{FS})}{(\delta^{15}\text{N}_{\text{TF}} \times \text{TFN})} \quad (5a)$$

$$P^{15}\text{N}_{\text{FR}} = 1 - P^{15}\text{N}_{\text{FS}} \quad (5b)$$

This proportion of the total faecal ^{15}N excess in the slow fraction is then used to attribute the total ^{15}NR of FLS_{UM} and FLS_{HM} (as calculated from Eq. 1) to the ^{15}N recovery from the slow and rapid faecal fractions ($^{15}\text{NR}_{\text{FS}}$ and $^{15}\text{NR}_{\text{FR}}$) by solving Eq. 6a and 6b simultaneously.

$$\begin{aligned} ^{15}\text{NR}_{\text{FLS}_{\text{UM}}} = & ^{15}\text{NR}_{\text{FS}} \times P^{15}\text{N}_{\text{FS}_{\text{UM}}} + ^{15}\text{NR}_{\text{FR}} \\ & \times (1 - P^{15}\text{N}_{\text{FS}_{\text{UM}}}) \end{aligned} \quad (6a)$$

$$^{15}\text{NR}_{\text{FLS}_{\text{HM}}} = ^{15}\text{NR}_{\text{FS}} \times P^{15}\text{N}_{\text{FS}_{\text{HM}}} + ^{15}\text{NR}_{\text{FR}} \times (1 - P^{15}\text{N}_{\text{FS}_{\text{HM}}}) \quad (6b)$$

By using this method, the results from non-homogeneously labelled faecal slurry can still be analysed and the recovery of the slow and rapid faecal fractions can be calculated individually.

Apparent N recovery

The apparent N recovery (ANR) from slurry in herbage was calculated as:

$$\text{ANR}(\%) = \frac{(\text{Nuptake}_{\text{slurry}} - \text{Nuptake}_{\text{control}})}{\text{N}_{\text{applied}}} \times 100 \quad (7)$$

where N = total N taken up by herbage or applied in slurry, respectively.

^{15}N recovery in stubble and roots

Due to repeated harvests in the experimental design, the recovery of ^{15}N in the stubble (<5 cm) and roots was not measured. The ^{15}N recovery in the stubble was estimated based on the recovery in stubble (as a % of the recovery in the harvested herbage) as measured in another experiment applying the same labelled slurries to soil cores which were harvested at similar dates (Hoekstra et al., unpublished data). Similarly, data from Whitehead and Bristow (1990) on the ^{15}N recovery of urine applied to a permanent sward were used to estimate the ^{15}N recovery in roots as a function of the ^{15}N recovery in the above-ground herbage and time after application.

^{15}N lost through ammonia volatilisation

The proportion of total ammoniacal N volatilisation was estimated using the ALFAM model (Søgaard et al. 2002). The variables included in the model were soil moisture content, air temperature, wind speed, manure type, dry matter content and ammoniacal nitrogen content of manure, application method and application rate (Søgaard et al. 2002). The model was run with the slurry composition data in Table 1, the weather

conditions during 24 h after application (mean temperature 15.7°C, average wind speed 3.4 ms⁻¹); the measuring technique was set to micro-meteorological mass balance, the soil moisture content to dry soil, and application method to broadcast application.

Statistical analysis

Statistical analysis was carried out using PROC MIXED in SAS. The fixed factors were slurry labelling method and harvest number. The experiment was corrected for repeated measures (harvests) by including a heterogeneous first-order autoregressive variance covariance structure in the model.

Results

Slurry composition and weather conditions

On average, the DM content of the slurry was 65 g kg⁻¹ and the N content was 84 g kg⁻¹ DM, with NH₄-N making up 55% of the total slurry-N (Table 1), which is within the range reported for cattle slurry. The slurry ^{15}N enrichment was 0.369, 0.937, 0.563 and 1.140 atom% for NLS, ULS, FLS_{UM} and FLS_{HM}, respectively. For FLS_{UM} and FLS_{HM}, both the slow and the rapid faecal fraction were enriched, but the slow fraction was less enriched than the rapid fraction (Table 1). Therefore, only method 2 (Eqs. 4, 5a and 5b) was suitable for calculating the ^{15}NR of the slow and rapid faecal fractions.

The weather conditions during the growing season in 2008 were extremely wet, and the mean daily rainfall during the last harvest interval was over twice the 30-year average for the period. As a result, the calculated excess drainage was 3 mm day⁻¹ during the last harvest interval in 2008, which was four times higher than in the same period in 2007 (Table 2).

Herbage dry matter yield and N uptake

The herbage dry matter yields (DMY) were on average 4.5, 1.4, 0.8, 7.3 and 2.9 t ha⁻¹ for the harvests at weeks 6, 12, 38, 53 and 62, respectively (data not shown). At week 6, the DMY for the control plots were significantly ($p < 0.001$) lower compared to

the plots with slurry applied (2.4 versus 5.0 t ha⁻¹), but there were no significant differences in DMY in the remaining harvests. There was no significant effect of slurry application on herbage-N concentration, and therefore the herbage-N uptake followed the same temporal patterns as DMY (data not shown). The ANR was on average 21% after 6 weeks and the residual ANR (weeks 12–62) was 4.8%, but the variation was very large, particularly for the residual cuts (data not shown).

Recovery of slurry ¹⁵N in herbage (¹⁵NRH)

There was a significant interaction ($p < 0.0001$) between harvest and slurry fraction on ¹⁵NRH (Fig. 3a). At week 6, the ¹⁵NRH of urine (U), the rapid faecal fraction (FR) and the slow faecal fraction (FS) was 18.3, 12.3 and 2.7%, respectively. The ¹⁵NRH of U decreased to 2.2% at week 12 and remained on average 0.8% for the later harvests. The ¹⁵NRH of FR decreased to on average 1.8%, and there were no significant differences between subsequent harvests. For FS, the ¹⁵NRH was similar across harvests and was on average 1.7%.

In order to calculate the contribution of the three fractions to the total slurry ¹⁵NRH, the ¹⁵NRH of the three fractions were multiplied with their relative proportions in the slurry total N (62, 25 and 13% for U, FR and FS, respectively) (Table 1 and Fig. 3b). There was a significant ($p < 0.0001$) effect of harvest on total slurry ¹⁵NRH which was highest at 6 weeks (14.7%), and declined to 2.1%, 0.9%, 1.6% and 0.8% at 12, 38, 42 and 53 weeks, respectively. There was a significant interaction ($p < 0.0001$) between harvest and slurry fraction on the contribution of the different fractions to total slurry ¹⁵NRH. At 6 weeks, approximately 78% of the total slurry ¹⁵NRH was from U,

20% from FR and 2% from FS, whereas from 38 to 62 weeks, on average 40% of total slurry ¹⁵NRH was from U and the remainder from FR and FS.

Recovery of slurry ¹⁵N in soil

¹⁵N recovery in soil total N (¹⁵NRS_T)

The total soil N content varied from 3.0 g kg⁻¹ dry soil in layer a (0–5 cm depth) to 1.5 g kg⁻¹ dry soil in layer c (15–25 cm depth) (data not shown).

There was a significant ($p < 0.001$) slurry fraction × depth × time interaction for ¹⁵NRS_T (Fig. 4).

The ¹⁵NRS_T from U summed over the three layers was 11.5% after 6 weeks, increased up to 20.7% after 53 weeks, and declined to 17.3% after 62 weeks, but these changes were not significant. During the first three harvests, most of the ¹⁵N was recovered in layer a, but the recovery in layer b increased significantly ($p < 0.0001$) with time, and after 62 weeks layer b contained about 50% of the ¹⁵N recovered in soil. The recovery in layer c did not change significantly over time and was on average 8% of the ¹⁵NRS_T.

For FR, the ¹⁵NRS_T increased from 33% after 6 weeks to 55% after 53 weeks, after which it declined to 34% at 62 weeks. Similar to the ¹⁵NRS_T recovery from U, the relative recovery in layer a was largest at 6 weeks, and decreased with time, whereas the relative recovery in layer b increased and the recovery in layer c remained fairly stable at, on average, 12% of ¹⁵NRS_T.

The ¹⁵NRS_T from FS was 15% at 6 weeks, increased to 65% after 53 weeks and decreased (non-significantly) to 50% after 62 weeks. During the first two harvests, no ¹⁵N was recovered in layer b and layer c, but for the last 3 harvests the proportion

Fig. 3 **a** The ¹⁵N recovery (%) of urine-N (U, white bars), rapidly degradable faecal-N (FR, grey bars) and slowly degradable faecal-N (FS, black bars) in herbage at 6, 12, 38, 53 and 62 weeks after application, and **b** the total slurry ¹⁵N recovery in herbage and the contribution of U, FR and FS. Error bars = 2 × SE ($n = 5$)

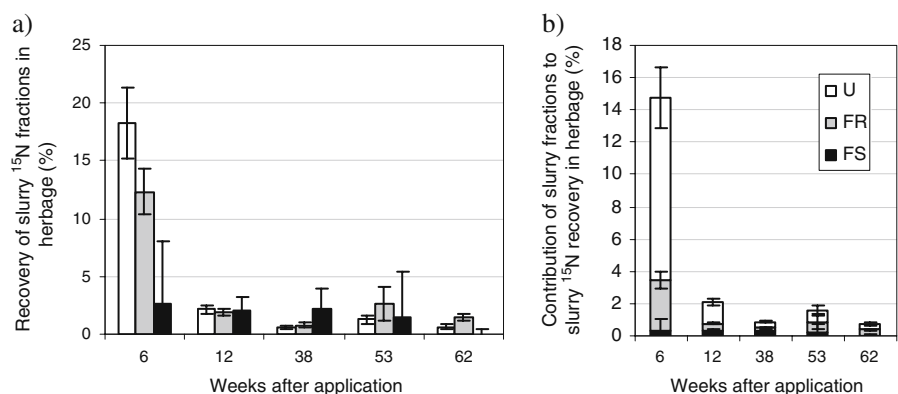
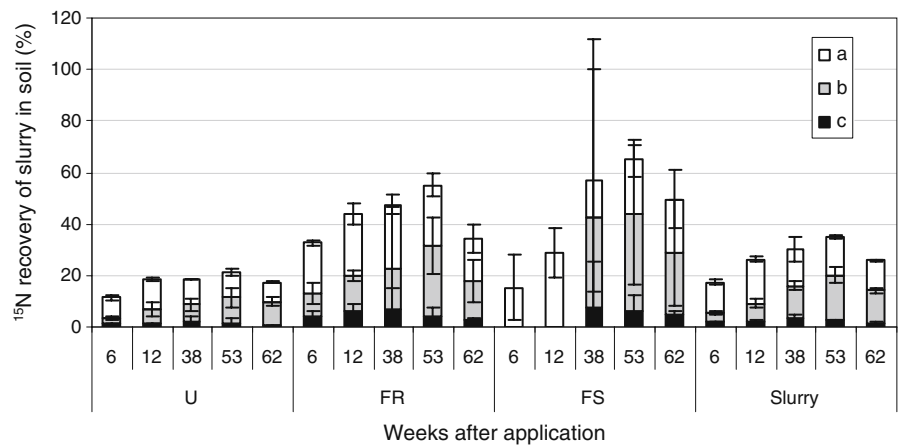


Fig. 4 The ^{15}N recovery (%) of urine-N (U), rapidly degradable faecal-N (FR), slowly degradable faecal-N (FS) and total slurry-N (calculated from weighted fractions) in the total soil N for three soil layers (a=0–5 cm, b=5–15 cm and c=15–25 cm) at 6, 12, 38, 53 and 62 weeks after application. Error bars= $2\times\text{SE}$ ($n=5$)



of $^{15}\text{NRS}_T$ recovered was 34%, 56% and 11% for layer a, b and c, respectively.

There was a significant effect of harvest on the calculated recovery of total slurry-N in soil ($^{15}\text{NRS}_T$ of fractions multiplied by the relative proportion of fraction in total slurry-N), which increased from 15% at 6 weeks to 35% after 53 weeks and declined to 26% after 62 weeks. There was a significant ($p<0.001$) interaction between layer and harvest, and the relative recovery in layer a decreased from 69% at 6 weeks to 53% at 62 weeks, whereas the relative recovery in layer c was stable at on average 9% of $^{15}\text{NRS}_T$.

^{15}N recovery in soil inorganic-N ($^{15}\text{NRS}_I$)

Soil mineral N content varied over the different harvests but showed no significant treatment effect. $^{15}\text{NRS}_I$ was very small, and represented on average <1% of $^{15}\text{NRS}_T$. There were no significant treatment effects (data not shown).

^{15}N recovery in soil microbial biomass ($^{15}\text{NRS}_{SMB}$)

On average 4% of the total soil N was in the form of SMB (data not shown). After 6 weeks, on average 4.6% of ^{15}N was recovered in the SMB (summed over 3 depths), and this declined to 2.3% after 62 weeks (Fig. 5). After 6 weeks the $^{15}\text{NRS}_{SMB}$ tended to be higher for U and FR compared to FS, but this difference between slurry fractions was not significant. There was a significant ($p<0.05$) interaction between harvest week and depth. After 6 weeks $^{15}\text{NRS}_{SMB}$ was significantly higher in layer a compared to b and c,

whereas after 62 weeks it was higher in layers a and b than in c.

After 6 weeks $^{15}\text{NRS}_{SMB}$ represented 45%, 22% and 11% of the total ^{15}N recovered in soil for U, FR and FS, respectively, and after 62 weeks this had decreased to 10%, 11% and 2%, respectively.

Estimated recovery in stubble and roots

The estimated ^{15}N recovery in the stubble decreased over time from 10.4% at week 6 (U) to <0.4% at

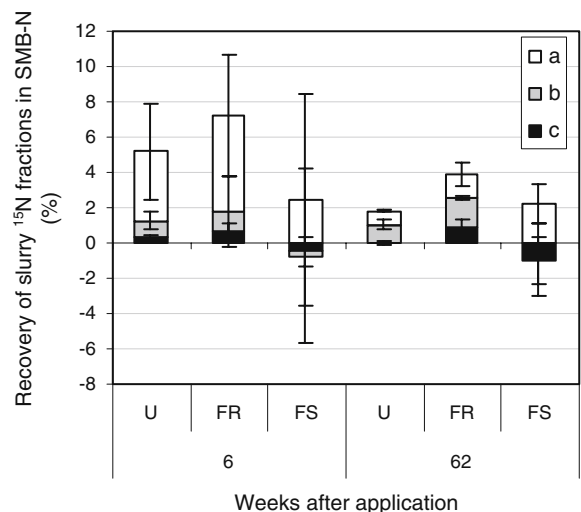


Fig. 5 The ^{15}N recovery (%) of urine-N (U), rapidly degradable faecal-N (FR) and slowly degradable faecal-N (FS) in the soil microbial biomass (SMB) for three soil layers (a=0–5 cm, b=5–15 cm and c=15–25 cm) at 6 and 62 weeks after application. Error bars= $2\times\text{SE}$ ($n=5$)

week 62 (FR and FS) (Fig. 6). A similar trend was found for the calculated ^{15}N recovery in the roots, which ranged from 5.2% at week 6 (U) to 1.2% at week 62 (FS) (Fig. 6).

The fate of slurry ^{15}N

There was a significant ($p < 0.0001$) harvest \times fraction interaction for the sum of the ^{15}NR in herbage (cumulated over 5 harvests), soil, roots and stubble ($^{15}\text{NR}_{\text{SUM}}$). For U, $^{15}\text{NR}_{\text{SUM}}$ was on average 46% and did not change significantly between harvests (Fig. 6). The predicted ammonia volatilisation accounted for another 46%, thus total urine- ^{15}N recovery was $>90\%$. For FR, the total ^{15}NR increased from 58% at 6 weeks to 75% at 53 weeks, after which it declined to 57% at 62 weeks. For FS, the initial recovery at 6 weeks was only 21%, the recovery increased sharply to 75% at 53 weeks and declined to 59% at 62 weeks.

On average, 51% of slurry-N could be accounted for in $^{15}\text{NR}_{\text{SUM}}$, whereas another 28% was predicted to be lost through ammonia volatilisation. Therefore, on average, 21% of slurry-N remained unaccounted for.

Discussion

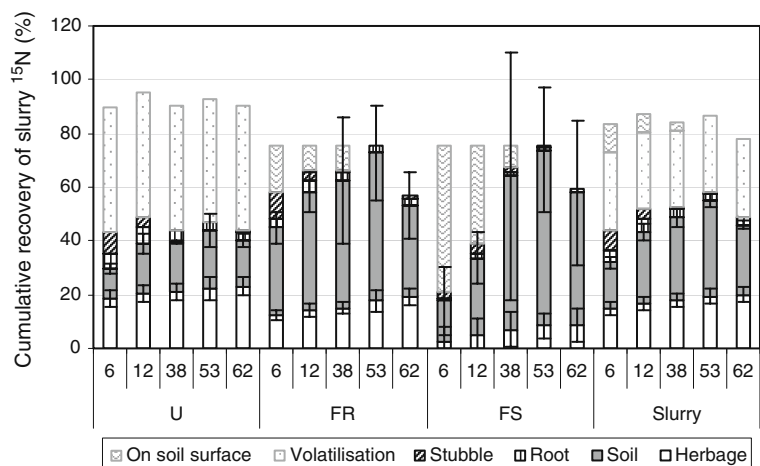
Labelling and calculation method

In this study, the ^{15}N herbage feeding method and the ^{15}N urea feeding method (Powell et al. 2004) were used to produce ^{15}N enriched faeces. The method was

thought to produce homogeneous labelling by recombining equal amounts of faeces around the peak ^{15}N enrichment, but this was not observed. The lower ^{15}N enrichment of the NDFN fraction in the FLS_{HM} may have been partly due to the enrichment of the herbage NDFN fraction, which tends to be lower than the enrichment of the grass cell contents (Bosshard 2007). Additionally, the NDFN fraction of the FLS_{SUM} was also enriched contrary to the findings of Powell et al. (2004), who suggested that only the rapidly available fraction would be enriched.

Therefore, we developed a new calculation method applying the theory by Sørensen et al. (1994) that divides faecal-N components into a slowly-decomposable pool consisting of undigested feed N and microbial N from the rumen, and a more rapidly decomposable pool consisting of N compounds in living microbes, partly-decomposed microbial tissues, digestive secretions and dead cells from tissues in the digestive tract (Fig. 2). As we had data for two differentially labelled slurries, both containing a labelled slow and rapid faecal-N pool, we could calculate the recovery of the two faecal fractions separately by comparison (see ^{15}N recovery of labelled fractions and Eqs. 4, 5a, 5b, 6a, 6b). There are two major advantages to this method: Firstly, we could accurately calculate the recovery of slurry that is not labelled homogeneously, and secondly, for the first time we were able to calculate the recoveries of three slurry-N pools (U, FR and FS) and, based on that, the total slurry ^{15}N recovery. The main downside of this method is a potential increase in the error, because two measurements are compared, as opposed to a direct calculation of the ^{15}NR .

Fig. 6 The fate of ^{15}N (%) of urine-N (U), rapidly degradable faecal-N (FR), slowly degradable faecal-N (FS) and total slurry-N (calculated from weighted fractions), in herbage (cumulative), soil (0–25 cm), roots (calculated), stubble (calculated), ammonia volatilisation (predicted by ALFAM model), and on the soil surface (estimated) at 6, 12, 38, 53 and 62 weeks after application. For measured data, error bars = $2 \times \text{SE}$ ($n=5$)



Fate of slurry-N in herbage

The total slurry ^{15}N in herbage was 15% after 6 weeks, which is lower than the ANR of 21%, indicating a substitution of tracer ^{15}N , resulting in a potential underestimation of the slurry-N recovery in the herbage with the ^{15}N method (Dittert et al. 1998; Schröder 2005a). However, direct comparison of these results requires caution, because the ANR is based on the mean of the three different slurries, whereas the ^{15}N is based on the weighted recovery of the labelled fraction in each slurry.

As expected, the ^{15}N in herbage 6 weeks after application was highest for the urine fraction (18.3%) followed by the FR (12.3%) and FS (2.7%), reflecting the plant availability of the three slurry fractions. The 18% recovery from the urine fraction is similar to recoveries reported for broadcast summer applied slurry by Hoekstra et al. (2010) using slurry of which the $\text{NH}_4\text{-N}$ pool was labelled with ^{15}N . There was a large difference between the recovery of the slow and the rapid faecal fractions, confirming that the two N pools had distinctly different N availabilities.

The total slurry ^{15}N recovery in herbage of 15% after 6 weeks was in close agreement with the apparent N recovery for broadcast slurry applied in June as reported by Lalor et al. (in press), but was higher than the agronomic advice in Ireland (Nitrogen Fertiliser Replacement Value [NFRV]=5%) (Coulter 2004). It should be noted that the NFRV is a ratio of recoveries, i.e. the recovery of slurry-N over the recovery of mineral fertiliser N, as opposed to N recovery. Therefore, the discrepancy between our results and those of Coulter is likely to be even larger (assuming a mineral fertiliser recovery of <100%).

The residual recovery in herbage (cumulated over the remaining 4 harvests) was 4.7%, 6.8% and 5.7% for U, FR and FS, respectively, indicating that the residual recovery of the faecal fractions is slightly higher than for urine-N, which is in agreement with other studies (Bosshard et al. 2009; Sørensen et al. 1998; Thomsen et al. 1997). The main difference between the residual effect from urine and faecal-N is that for the faecal fractions, the residual effect is mainly the result of gradual mineralisation of organic N, whereas for U it is caused by the gradual mobilisation of inorganic-N immobilised shortly after application (Jensen et al. 2000; Sørensen and Amato 2002).

Fate of slurry-N in soil

The recovery of urine-N and faecal-N in soil was comparable with results from other non-grassland based studies in which ^{15}N labelled manures were applied to soils (Bosshard et al. 2009; Sørensen et al. 1998; Thomsen et al. 1997).

The recovery of the faecal fractions was variable, with a strong increase in recovery (particularly for FS) up to 38 weeks, followed by a decline after 53 weeks. This strong increase may be related to the incorporation of faecal matter that had previously remained in the stubble and on top of the soil after application. Our soil sampling method would not have accounted for this fraction, as cores were taken from the bare soil surface between grass tillers, and stubble residues were removed from the samples before sieving. It is estimated that (Fig. 6, on soil surface) 6 weeks after application, over 70% and 20% of the slow and rapid faecal fractions, respectively, had not been incorporated into the soil, equating to just over 10% of total slurry-N. The fact that it took 38 weeks (the winter period) before FS was recovered in the deeper soil layers, would confirm the slow incorporation of the more fibrous slurry component into the soil.

The decrease in slurry-N recovery in soil after 53 weeks may be related to the excessive rainfall and resulting net drainage during this period (Table 2), which may have resulted in increased leaching (of both nitrate and dissolved organic N) (Van Kessel et al. 2009; Wachendorf et al. 2005) or enhanced loss of N as N_2 and N_2O due to higher denitrification rates (Jahangir et al. 2010).

After 6 and 62 weeks, <1% of slurry ^{15}N was recovered in the inorganic soil N pool (KCl-extract), which is in line with previous work (Hoekstra et al. 2010; Morvan et al. 1997) and is due to the rapid uptake into herbage and immobilisation of mineral N in soil.

Immobilisation of $\text{NH}_4\text{-N}$ in soil after fertiliser application can vary considerably, depending on slurry composition, placement, soil and crop type and temperature (Jackson et al. 1989; Ledgard et al. 1989; Morvan et al. 1997; Petersen 2006). The proportion of slurry $\text{NH}_4\text{-N}$ (urine fraction) immobilised after 6 weeks in this study was relatively low at <5% (Fig. 5), compared to reported slurry $\text{NH}_4\text{-N}$ recoveries in SMB in grassland of 15% (Morvan et al. 1997) up to 60% (Jackson et al. 1989). This could be related to the relatively large losses of $\text{NH}_4\text{-N}$ through volatilisation.

When the urine-N recovery in SMB was expressed as a percentage of the ^{15}N recovered in total soil, the recovery was 45%, which is more in line with these other studies. Even though there was no significant difference in $^{15}\text{NRS}_{\text{SMB}}$ as a percentage of total ^{15}N applied, the proportion of soil ^{15}N recovered in the SMB was lower for the faecal fractions compared to urine (on average 20% and 45%, respectively). This is to be expected because faecal (organic) N is more resistant to microbial degradation. We have been unable to identify any other studies that have investigated the recovery of the faecal slurry fraction in SMB-N.

Cumulative recovery and losses

The ^{15}N recovery in the roots and stubble were not measured in this experiment, however, calculations based on recoveries reported in the literature (Whitehead and Bristow 1990) and from related experiments (Hoekstra et al., unpublished), suggested that even though roots and stubble were significant sinks for ^{15}N during the initial phase of the experiment (up to 13% of total slurry-N, Fig. 6), the contribution after 62 weeks would have been very small (3.3% of total slurry-N).

When adding up the total recovery of U, FR and FS over all the measured (herbage and soil) and estimated (stubble, roots, on top of soil) sinks, the recovery was on average 45% for U and 72% for FR and FS. The U not accounted for can largely be attributed to losses through ammonia volatilisation after application (Fig. 6). The remaining 10% could have been lost through leaching and denitrification (Schulte et al. 2006). The faecal-N not accounted for is harder to explain and may be attributed to losses through mineralisation followed by leaching or denitrification. Additionally, some faecal-N was likely incorporated into the soil faunal biomass. For example, earthworm populations, which had a biomass of up to 189 g m^{-2} in a pasture at the same research station (Curry et al. 2008), have been shown elsewhere under field conditions to assimilate slurry-derived N (Schmidt and Ostle 1999). Transport of faecal-N below the sampling depth (25 cm) by deep-burrowing earthworms such as *Lumbricus terrestris* or through their vertical channels, could also have occurred.

Implications and future work

Results indicated that the bulk of the slurry-N recovery in herbage (approximately 17% of slurry-N

applied) after a June application occurs in the year of application, with only 3% recovered in the subsequent year. This 3% (equivalent to 5 kg N ha^{-1}) may seem negligible from an agronomic point of view. However, it is crucial to consider that, with the practice of yearly repeated slurry application, as is customary in winter housing grassland systems, this small effect may add up to significant cumulative effects (e.g. Schröder 2005b).

However, a 20% recovery of slurry N in herbage over 2 years is still quite low compared to countries such as The Netherlands and Denmark, where NFRV's for cattle slurry on grassland are set at 50 and 70%, respectively and there is pressure on Ireland to reach the same targets.

It should be noted that the conditions and methods of slurry application during this experiment were not aimed at optimising the slurry N recovery. Research has shown that slurry N utilisation is higher for spring applied slurry (as opposed to summer applied) and band application or injection (as opposed to broadcast) (Hoekstra et al., 2010; Lalor et al. 2009, *in press*) due to a combination of better crop utilisation and lower losses through ammonia volatilisation. Currently, best practice in Ireland is to apply slurry with a trailing shoe in spring time, and this could increase the N recovery in the first year up to at least 25% (Lalor et al. *in press*). Also, the weather conditions during 2007, but particularly late summer 2008 were exceptionally wet, which may have resulted in an N recovery which is lower than could be expected in other years. Additionally, the isotope substitution effect may have resulted in a slight underestimation of the slurry N recovery in herbage. However, based on current Irish studies, it is unlikely that NFRV's of over 40% are achievable in practice under current Irish conditions.

Slurry is notorious for its variable composition, which may be the result of differences in animal type, animal feeding, slurry storage, etc. In this study, we have distinguished between three separate slurry fractions for the first time. Further work is needed to investigate whether we can establish a link between the recovery of these different slurry fractions and slurry composition (e.g. NIRS, structural and chemical analysis), in order to improve the prediction of the recovery of slurry-N from various sources.

Conclusions

- A new calculation method was developed that can be used to quantify the recovery of both the slow and rapidly available faecal-N fractions based on the combined results from the herbage and urea ^{15}N labelling methods. This method has the important benefit that results from non-homogeneously labelled faecal-N can be correctly interpreted. By applying this method, the N recovery from three different slurry-N fractions (urine, rapid faecal and slow faecal) could be distinguished for the first time.
- The initial recovery (6 weeks after application) in herbage was 18%, 13%, 2% for U, FR and FS, respectively, and the residual recovery (12–63 weeks after application) was 4%, 6% and 7% for U, FR and FS, respectively.
- The total recovery in the plant-soil system ranged from 45% for urine-N to 72% for faecal-N.
- The ^{15}N results in combination with results of agronomic studies will form the basis for models predicting both the initial and residual nitrogen recoveries from cattle slurry on grassland.

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