Long term fate of slurry derived nitrogen in soil: A case study with a macro-lysimeter experiment having received high loads of pig slurry (Solepur)

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Abstract

In intensive livestock production areas, land application remains the traditional management of manure and slurries for nutrient recycling. For sustainable agriculture there is fear, however, that this practice may have detrimental effects, particularly on the depletion of Soil Organic Matter associated with pig slurry applications. We investigated the long-term fate of nitrogen in a reconstituted soil having received high doses of pig slurry during 5 years (1991–1995). After 5 years of intensive application rates (nearly 1000 m³ yr⁻¹), the N and C content of the soil profile (0–20 cm) had increased by about 60% and 50%, respectively. These results confirm previous findings although it seems that the particularly high rates of application may explain, in part, the relatively important N incorporation in soil. Pig slurry applications ceased in 1995 and nitrogen content in soil and drainage water have been monitored. Apparent mineralization rates were calculated from the decrease in N content of the soil. This analysis indicated that more than 50% of the added N stored in the soil at the end of the applications would eventually be mineralized, leaving nearly 50% of the stored N to be immobilized in the soil. These results are the first published of their kinds, as most reports never examine the fate of applied pig slurry N after halting applications. In addition the few reports on long-term experiments suggest that Soil Organic Matter following pig slurry applications may be unstable. Our analysis tends to show the contrary. However, this conclusion must be tempered because data on nitrate leachate patterns suggest that soil management such as ploughing and sowing may actually trigger mineralization that could eventually deplete nitrogen stored following applications.

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1. Introduction

A new framework policy to promote soil protection and sustainable land use has been developed by the Environmental Action Programme of the European Union (EU) (Quevauviller and Olazabal, 2003). This EU strategy comprises eight major issues including erosion, decline in soil organic matter content, compaction, contamination, loss of biodiversity, sealing, landslides and flooding. This policy will require consultation and experimental data sets for the examination of soil quality (Quevauviller and Olazabal, 2003).

In intensive livestock production areas, land application remains the traditional management of manure and slurries for nutrient recycling. This management may have both positive and negative consequences for soil quality. Indeed, these practices have resulted in a build up of major nutrients (such as nitrogen, phosphorus, potassium) and micronutrients (such as copper and zinc) into soil profiles (Coppenet et al., 1993; Leinweber et al., 1997). This in turn increases the risk of nutrient loss to receiving water bodies or aquifers through runoff and leaching (Schröder, 2005). On the positive side, manure addition to soil theoretically has the potential to increase organic matter content to
soils, which would be of great value for sustainable agriculture. It is unclear, however, whether manure or slurry addition to soil does have a long term positive effect on organic matter content in the long run. Similarly, there is a lack of knowledge on the fate of immobilized nitrogen initially added in manure, in the long term.

In this paper we present results from a reconstituted soil or macro-lysimeter experiment initially designed as a treatment reactor (Martinez, 1997) having received high doses of pig slurry during 5 years (1991–1995) and monitored during the ten subsequent years. The aim of the paper is to quantify the long term fate of the applied pig slurry nitrogen and to examine its stabilization over a ten year period.

2. Methods

2.1. Plant construction and effluent application

The study site is located in Western Brittany near the town of Plouvorn (France, 48°58’ N; 4°03’ W). The climate is humid and temperate with a mean annual rainfall of 1100 mm and a mean temperature of 11 °C (n = 36). This experimental facility (macro-lysimeter) consisted of a managed field of 3280 m² in size, 80 cm deep, hydrologically isolated, which allowed collection of all the water leaching through the reconstituted soil profile on which ryegrass (Lolium perenne sp.) was grown. The soil is silty loam with a soil texture of 15% clay, 58% silt and 27% sand (Fardeau and Martinez, 1996). From 1991 to 1995, thirty repeated doses of raw pig slurry were applied on this managed field. Table 1 shows the total carbon and nitrogen loads applied during this intensive spreading period. Further complementary details are given in a previous paper (Martinez, 1997).

From 1996 to 2005 this experimental treatment facility was maintained and no more pig slurry was applied. This was done so as to study the fate of the accumulated soil N over time. Ryegrass sward was maintained over the lysimeter and regularly cut and left in place so that there would be no plant exportation. In October 1995, September 1999 and September 2003, the field was ploughed to a depth of 20 cm and sown back to ryegrass again. All drainage water was collected by artificial drains positioned at the bottom of the lysimeter.

2.2. Soil and water sampling

For soil sampling, the experimental lysimeter was divided into four equal plots of approximately 800 m² in size. These four plots were sampled at 0–20, 20–40 and 40–60 cm depths. Nine soil cores (5 cm, diameter) were sampled for each layer and then sieved and mixed through a 6 mm mesh. A sub-sample of about 1 kg of fresh soil was taken for each depth.

During the first phase of the experiment, soil samples were taken on the following dates: 19 March 1991, 17 March 1992, 16 March 1993, 19 April 1994, 30 March 1995 and 4 April 1996. After manure applications ceased, the soil sampling campaign was carried out on 3-year intervals: 15 March 1999, 5 April 2002 and 24 March 2005.

Soil bulk density was measured in 1991 in the Solepur macrolysimeter from undisturbed samples obtained from a soil pit for the three soil layers explored. An average of 1.3 kg L⁻¹ for all layers was found and used in this study.

During the drainage seasons (October–April in general), drainage flow rates were measured using a narrow V-notch weir equipped with an ultrasonic water level recorder (ISCO 3210, USA). Drainage water was sampled four times a day and composted into a 1-L bottle using an automatic sampler (ISCO 2100, USA). Samples were then collected on a biweekly basis during site servicing.

2.3. Laboratory analyses

Soil sample were first oven-dried at 45 °C, then were finely ground before analyses. Determination of total carbon was realized by sulfochromic oxidation (AFNOR (b), 1998) and soil total nitrogen was analyzed using a modified Kjeldahl method (AFNOR (a), 1995).

Throughout the drainage season, drainage water samples were analyzed for nitrate (NO₃⁻) concentration by ion chromatography (Dionex, USA). This procedure was initiated during the 1993–1994 drainage seasons and continued until May 2005.

3. Results and discussion

3.1. Nitrogen pig slurry loading

Chemical properties of the pig slurry applied (Table 1) during the loading period (1991–1995) were similar to those observed by others (e.g. Bernal et al., 1993; Scotford et al., 1998). On average over the 5 years, the slurry applied contained 57 kg m⁻³ of total solid and 23 kg m⁻³ of total carbon, 3.2 kg m⁻³ of total ammoniacal nitrogen and 5 kg m⁻³ of total Kjeldahl nitrogen. The C:N ratio of the slurries applied to the soil were close to 5, which falls within the range of commonly reported values for slurries (C:N from 3 to 6; e.g. Chantigny et al., 2004; Scotford
et al., 1998; Shi et al., 2004; Sorensen and Thomsen, 2005). Using the solid fraction of the pig slurry, thus removing ammonium which comprises a large proportion of the N content in the material from the calculation, the C:N ratio reached 13.8 (Table 1). This value is well within the reported range of animal manure C:N ratios (C:N from 13 to 16; e.g. Cordovil et al., 2005; Serna and Pomares, 1991; Shi et al., 2004; Sorensen, 2001).

3.2. Nitrogen accumulation in soil

From 1991 to 1996, during the intensive application period, total soil nitrogen content increased from 1.8 to 3.5 g kg\(^{-1}\) for the top layer (0–20 cm), 1.4–2.0 g kg\(^{-1}\) for the 20–40 cm layer and 0.7–0.9 g kg\(^{-1}\) for the 40–60 cm layer (Fig. 1). At the end of the application period, 6084 kg of total nitrogen were estimated to have been retained in the soil profile (0–60 cm), which represented about a quarter of the total N applied (24 t ha\(^{-1}\)) (Fig. 1). Previous nitrogen balances from the Solepur process have highlighted that a relatively small portion of the total N applied was recovered as NO\(_3\) in drainage water (12%; Martinez and Peu, 2000). More than 60% of the total N applied has not been accounted for either in the soil or the drainage water. These losses have been attributed in part to ammonia volatilisation using results from Chadwick et al. (1998), although this process probably accounted to hardly more than 10% of the total N (Chadwick et al., 1998). More likely, most of the losses could probably be attributed to denitrification (Chadwick et al., 1998).

During 5 years of intensive applications, the soil total N stock in soil increased by about 60% (from 10 to 16 t ha\(^{-1}\)), carbon content increased by 49% (from 102 to 153 t ha\(^{-1}\)). These results clearly illustrate that application of pig slurry can increase, at least temporarily, soil carbon and nitrogen contents. Soil organic matter (SOM) was not measured in this experiment but the increase of both carbon and nitrogen contents strongly suggest that SOM probably increased in the mean time.

These results confirm a number of previous reports. Hountin et al. (1997) found that after 14 years of pig slurry applications, the total N content had increased 1.6-fold in their studied soil. Paustian et al. (1992) similarly found that with organic amendments (green manure and different types of farm manures), the percentage of active soil organic matter increased (about 10%) during the first 10 yr after the additions were started and subsequently remained fairly constant. Similar results were observed by Bernal et al. (1993) with smaller application rates. However, the authors found that pig slurry application rates required to produce a significant soil N increase had to be higher than 300 m\(^3\) ha\(^{-1}\), which correspond to higher rates than commonly applied (ca. 50 m\(^3\) ha\(^{-1}\)). Other authors reported no or negligible enhancement of organic C in amended soils (Rochette et al., 2000). Plaza et al. (2005) actually found that in semiarid conditions, pig slurry applications to soils could actually trigger partial mineralization of native soil organic C through extended microbial oxidation. These results were obtained for 30–150 m\(^3\) ha\(^{-1}\) yr\(^{-1}\) application rates. In light of the results above, it is possible that our observations may be linked to the uncommonly high application rates (nearly 1000 m\(^3\) ha\(^{-1}\) yr\(^{-1}\); Table 1).

Nitrogen soil data recovered during our experiment only included total nitrogen and mineral and organic species were not specifically measured (Martinez, 1997; Martinez and Peu, 2000). It is thus unclear whether the increase in total N corresponded to an increase in the mineral N or organic N or both. Several authors have pointed out that this total nitrogen accumulation could be the result of ammonium immobilization (e.g. Chantigny et al., 2004; Trehan, 1994) or accumulation by rapid incorporation of slurry ammonium into an organic form (e.g. Chantigny et al., 2004; Morvan et al., 1997; Munoz et al., 2003; Sorensen and Amato, 2002; Sorensen and Jensen, 1995). However, accumulation could also be explained by the incorporation of organic nitrogen fraction of slurry. Using \(^{15}\)N-labelling techniques, Sorensen (1998) was able to show that a large proportion of the applied organic N was still in the soil, even after 18 months of decomposition.

For the special Solepur study case, the source of total nitrogen accumulation into the profile was not clear, nevertheless, both organic N load applied (8303 kg) and ammoniacal N load (16233 kg) could count for the measured soil N increase.

3.3. Apparent nitrogen mineralization

After the slurry applications ceased, a decrease in total soil N content was expected due to the mineralization of the easily degradable SOM recently constituted by pig slurry spreading, as well as leaching of stored mineral nitrogen (processes reviewed by Jarvis et al., 1996).

Between 1996 and 2005, the total soil N content decreased significantly in the top layer from 3.5 to 2.5 g kg\(^{-1}\) (Student t-test, \(p < 0.05\)). No significant varia-
tions (Student t-test, \( p < 0.05 \)) were recorded for the deeper 20–40 cm and 40–60 cm layers, respectively (Fig. 1). By analogy with published data (Cordovil et al., 2005; Dou et al., 1996; Stanford and Smith, 1972), we considered this decrease to be the equivalent of mineralization processes, although the actual processes listed above may not strictly correspond to what is usually referred to as mineralization (Jarvis et al., 1996). Two distinct decrease phases could be distinguished: a rapid decrease phase between 1996 and 2002 and a slower decrease from 2002 to 2005 (Fig. 1). By linear interpolation between consecutive N content measurements, apparent mineralization rates were estimated to be 556 kg N ha\(^{-1}\) yr\(^{-1}\) (1996–1999), 257 kg N ha\(^{-1}\) yr\(^{-1}\) (1999–2002) and 55 kg N ha\(^{-1}\) yr\(^{-1}\) (2002 and 2005) using a constant soil bulk density of 1.3 kg L\(^{-1}\). Bulk density was considered to be constant in this case study because the soil was not used for crop production. Indeed the field was initially seeded with a ryegrass crop cover only ploughed and reseeded in 1995, 1999 and 2003 to prevent adventitious plant expansion on soil. A sensitivity analysis (not presented) has further demonstrated that a possible decrease in bulk density (from 1.3 to 1.1 kg L\(^{-1}\)) induced by occasional ploughing could contribute to an underestimation of 10% of the N mineralization rates.

It is difficult to compare apparent N mineralization measured to data published as there are, to our knowledge, no reports describing the evolution of soil N content after halting of manure or slurry applications. In a recent article Schröder (2005) argued that “[...] The phenomenon of residual effects also implies that the long-term effects of reduced inputs only become fully visible after many years. Long lasting experiments are hence needed to approximate the true value of N fertilizer value of manure and to construct and verify appropriate models” [...] There are, however, numerous reports on mineralization rates obtained on a short-term basis from soil cores incubated in the laboratory. In these specific laboratory conditions, mineralization rate were higher than those observed in our study and were comprised between 10 and more than 1000 kg N ha\(^{-1}\) yr\(^{-1}\) (e.g. Cordovil et al., 2005; Dou et al., 1996). Our results seem to be lower than most mineralization rates reported, but this statement should be tempered because processes involved, the temporal and the spatial scale at which they were measured are very different.

From 1996 to 2005, the apparent nitrogen mineralization flux was estimated to have reached 2.6 t ha\(^{-1}\), which represents the disappearance of more than 43% of the N recently accumulated in the soil (6 t ha\(^{-1}\) of N accumulated between 1991 and 1996). Our results are somewhat worrying as the decrease did not seem to have ceased in 2005, thus suggesting that the actual N storage corresponded to a short term process rather than long-term immobilization of N in soil.

To explore whether stored N would eventually be totally released or, if there would be a soil N level at which apparent mineralization would be “halted”, we fitted an exponential curve on the amount of N apparently mineralized in the 0–20 cm horizon, for the period 1996–2005, following suggestion from Stanford and Smith (1972). Quantity of N mineralized was thus computed assuming that at any time \( t \), the cumulated mineralized N(\( t \)) could be written as:

\[
N(t) = N_0[1 - \exp(-kt)]
\]

where \( N_0 \) is the amount of N that would eventually be mineralized and \( k \) is the mineralization rate.

Based on a non-linear least square regression, model fitting yielded a mineralization potential for \( N_0 \) of 2811 kg N ha\(^{-1}\) and a constant mineralization rate of 0.302 yr\(^{-1}\). Comparison between the cumulative apparent N mineralization recorded into the Solepur field and the predicted cumulative apparent N mineralization indicate that this model produced good fit (\( R^2 = 0.91 \); Fig. 2). Removing 2811 kg N ha\(^{-1}\) from the total N soil content at the end of the applications yields the amount of N that would eventually remain in the soil. The results suggest that more than 47% of the pig slurry accumulated at the end of the application period, would remain in the soil. This result is very interesting and quite comforting as it suggests that intensive applications on our soil could store N in soil on a long-term basis.

To our knowledge, there are no reports of similar experiments where the fate of nitrogen was monitored after halting of pig slurry applications. Few N accumulation results from long-term experiments are reported in the literature and in all cases the manure or slurry were applied on a continuous basis and were not halted. Paustian et al. (1992) showed that after 30 years of applications, soil N contents receiving external organic-matter inputs (green manure and different types of farm manures) maintained or increased in soil N, while N declined in the three treatments receiving no organic amendments. Wander et al. (1994) investigating different organic management practices on soil fertility showed that after 10 years of amendments, the soils receiving the organic treatments accumulated biologically active

![Fig. 2. Non-linear least squares regression comparing observed (black circles) and predicted (line) cumulative mineralized N in the Solepur process (kg N ha\(^{-1}\)).](image-url)
carbon and SOM. Chang and Janzen (1996) measured, after 20 years of application of feedlot manure, higher organic soil N content for soils having received higher application rates. These papers all report an increase in soil N content after long term application of manure. It is not known, however, whether the increase corresponded to an overall increase of stable organic matter in soil. Paustian et al. (1992), suggested that the rate of C addition was the single most important factor determining organic-matter levels in their soils. Pig slurry containing relatively small amounts of C compared to N (C:N between 3 and 6), some authors have expressed concerns that applied slurry would tend to trigger mineralization of native SOM, rather than incorporating applied N in the soil (e.g. Plaza et al., 2004). Indeed, with “[…] easily decomposable amount of organic C and the relatively large N content of pig slurry applied to soil […] soil micro-organisms have an increased amount of fresh N available for their protein metabolism but not enough fresh C as the energy source, thus microbial oxidation of soil native organic C must occur” (Plaza et al., 2004). Accordingly, Rochette et al. (2000) measured no significant increase of soil C content after 19 consecutive years of pig slurry application at rates of 60 and 120 m³ ha⁻¹ yr⁻¹. The same authors found that there were no long-term effects of pig slurry application on the soil microbial biomass. Our results thus somewhat contradict previous research conducted after long term application of pig slurry on soils, and are closer to results obtained with manure. However, it should be noted that there was no crop export during the application period and it is possible that weeds and wild plants freely growing on the uncultivated soil added some organic C to the top soil. However, the quantities of C added were probably not high enough to significantly increase the C content of the top soil.

Additional information obtained from drained water nitrate gives more light on the actual dynamics of the processes and are discussed below.

3.4. Dynamics of nitrate leaching

Dynamics of nitrate leaching on a multi-annual basis is represented in Fig. 3. A linear relationship between a cumulative nutrient load and the cumulative drainage volume would indicate that the source of NO₃⁻ is seemingly unlimited. Indeed, regardless of the amount or the intensity of water leaching through a soil profile, that NO₃⁻ would be released at a rate sufficient to maintain the linear relationship. Leaching curves for the three slurry application periods are actually sigmoid in shape (Fig. 3). This suggests that the source or the ease at which nitrate “diffused” into leaching water actually increased during the course of a year. The increased slope of the curves actually corresponds to slurry application periods. The slope then regularly decreased to form the second part of the S shape. This suggests that the source of nitrate actually decreased as it took more water to export the same amount of nitrate.

After the pig slurry applications ceased, and during three years (1996–1997, 1997–1998 and 1998–1999), the slope of the curves regularly decreased, suggesting, in light of what is presented above, that the source of nitrate leaching actually decreased.

In 1996, ryegrass sward was sowed. It was then cut on a regular basis but grass was left in place. In 1999, the experimental plot was ploughed and sowed in grass to maintain the ryegrass sward and the same soil management was applied in 2003. It is remarkable to note that at each ploughing and sowing period corresponds a clear increase and a break in the slope of the multi-annual curves. This suggests that ploughing clearly increased the source of nitrate reaching leaching water.

Nitrate leaching losses in the Solepur lysimeter between 1999 and 2005 accounted to 1200 kg N ha⁻¹ suggesting that 46% of the apparent N mineralise is recovered into the drainage water. Total N losses in drainage water was negligible with N concentration up to 5 mg L⁻¹. Soil denitrification appears to be an important component of this budget removing about 54% of the apparent N mineralization (Martinez and Peu, 2000).

Recent results describing the dynamics of nitrate leaching associated with drainage have shown that nitrate export usually follows an asymptotic curve as a function of drained water, reflecting the apparent leaching of residual soil N (Arlot, 1999). Exponential models have been proposed to describe nitrate leaching (Arlot, 1999; Magesan et al., 1994; Scotter et al., 1993). We thus used this type of model to fit to our data. The flux of nitrate exported can be written as (Arlot, 1999):

\[ F_{NO_3^-} = S_0[1 - \exp(-D/a)] \]

where \( S_0 \) is the leachable initial nitrogen stock in soil (kg ha⁻¹), \( D \) is the drainage volume (mm) and \( a \) is a conceptual “leaching porosity” (Arlot, 1999). This model was fitted to the first three years after applications ceased and corresponded remarkably well with measured data.
suggesting that the processes driving exportation of nitrate corresponded well to the process described by the model. The leachable initial nitrogen stock found by best fitting the model was 891 kg N ha\(^{-1}\) while the conceptual "leaching porosity" was 308 mm. The first value shows the great leaching potential associated with the rates at which the applications were performed. Nearly 92% of the nitrate leaching occurred within the first three years after the cessation of applications.

We extrapolated the model results to the following years to evaluate the exportation of nitrate that would have been exported without new soil management ("model" curve in Fig. 3). The sudden divergence between the model and the measured curves show that ploughing and sowing, are clearly responsible for exportation of new nitrate that would not have been available for exportation without the soil management in 1999 and 2003. There is ample evidence that tillage tends to increase mineralization and nutrient export in soils (reviewed by Balesdent et al., 2000; Kuzyakov et al., 2000). One of the main reasons for these observations is that tillage exposes the organic matter that is physically protected in microaggregates from biodegradation in addition to organic matter from the incorporated vegetation.

It thus seems that most of the easily mineralizable organic matter in soil was exported within the first three years after cessation of application and that lower amounts of N would have been exported had the soil not been disturbed.

Our results from the previous section were obtained on soil N content actually obtained after (2002 and 2005) the two ploughing and sowing events (1999 and 2003). The apparent mineralization rates computed thus correspond to the combined mineralization of undisturbed soil and the mineralization induced by tillage. Our conclusion that a sizable amount of incorporated N was immobilized in a rather stable form may very well be invalid or only represent a particular soil management after application. Indeed, looking at the increased exportation of nitrate associated with ploughing, it seems that annual ploughing could very well trigger additional exportations of N that could eventually deplete N immobilized in the soil. Information brought by analysis of nitrate exportation dynamics thus showed that immobilization of applied slurry N to soil is totally dependent on soil tillage and management.

4. Conclusions

The long-term fate of nitrogen in a reconstituted soil having received high doses of pig slurry during 5 years (1991–1995) was monitored. After 6 years of intensive application rates (nearly 1000 m\(^3\) yr\(^{-1}\)), we were able to show that the N and C content of the soil profile (0–60 cm) had increased by about 60% and 50%, respectively. These results confirm previous findings although it seems that the particularly high rates of application may explain in part the relatively important N immobilization in soil. Since 1996, the pig slurry applications have been halted and nitrogen content in soil and drainage water have been monitored. Apparent mineralization rates were calculated from the decrease in N content of the top soil layer (0–20 cm) from 1999 to 2003 as the deeper layers did not exhibit significant changes. This analysis yielded that more than 50% of the added N stored in the soil at the end of the applications would eventually be mineralized, leaving nearly 50% of the stored N to be immobilized in the soil. These results are the first published of their kind, as most reports never examine the fate of applied pig slurry N after halting applications. In addition the few reports on long-term experiments suggest that Soil Organic Matter following pig slurry applications may be unstable. Our analysis tends to show the contrary. However, this conclusion must be tempered because data on nitrate exportation dynamics suggest that soil management such as ploughing and sowing may actually trigger mineralization that could eventually deplete nitrogen stored following applications.

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